The effect of wood ash on the soil properties and nutrition and growth of *Eucalyptus grandis x urophylla* grown on a sandy coastal soil in Zululand.

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DECLARATION

By submitting this thesis electronically, I declare that the entirety of the work contained therein is my own, original work, that I am the authorship owner thereof (unless to the extent explicitly otherwise stated) and that I have not previously in its entirety or I part submitted it for obtaining any qualification.

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Abstract

A field trial of six replications was established to test the effect of various wood ash and fertilisers application rates on soil chemistry, tree nutrition and early growth rate of a clonal *Eucalyptus grandis x urophylla* stand. Wood ash from pulpmills is currently disposed of in landfills. Increasing costs and potential environmental risks have driven companies to investigate alternative disposal methods. Ash consists of a combination of carbonates, hydroxides and other calcium containing minerals that induce the liming effect if ash is applied to a soil. The trial was established near Richards Bay in October 2013 on a sandy soil with a low buffer capacity and a pH of approximately 5.5. The trial consists of four wood ash application rates in combination with three levels of fertiliser, viz. no fertiliser, 150 g conventional NPK fertiliser mixture, or 320g NPK controlled release mixture. Fertiliser mixtures and application levels were based on previous fertiliser trials in the region. Ash application rates for the field trial were based on a lab incubation study done with soil samples from Richards Bay, to which increasing amounts of lime were added. The study tested wood ash application rates of 0, 300, 600 and 1200 kg/ha. Field measurements were taken at 4 and 8 months after trial establishment. The primary objective was to investigate which application levels in combination with the type of fertiliser could be applied to soils without negatively affecting the stand nutrition or increase the levels of potentially harmful elements in the soil; thus investigating the feasibility of safely disposing wood ash on plantation soils as an alternative disposal method.

Soil nutrient concentrations were not affected by individual wood ash treatments, but more a product of the time interval after the ash additions were made. Soil C, P, K^+ and Mg^{2+} showed decreased concentrations from 4-8 months after establishment. Ca^{2+} concentrations increased in the same time interval. In addition, Na^+ and B concentrations decreased from 4-8 months. Soil heavy metal concentrations for Cd, Hg, Cr and Pb, analysed for 0-1200 kg/ha ash rates, were well below toxic levels at both time intervals. Wood ash induced a temporary liming effect. Mean soil pH increased with 0.6 units for the period 0 - 4 months and decreased with 0.4 units at 4 - 8 months after trial establishment.

Foliar nutrient analyses and assessment techniques revealed sub-optimal nutrient concentrations for P, K and Zn at 4 and 8 months of age. Concentrations were defined as sub-optimal, as none of the nutrients were below critical levels. Foliar heavy metal concentrations for Cd, Hg, Cr and Pb, measured at both time intervals, were less than 1mg/kg. The small concentrations found in this project were attributed to the low bioavailability of all four elements and were likely a product of the edaphic factors at Richards Bay, which was representative of a large greater portion of the Zululand coastal plain sites. The response in biomass index ranged between 13 % and 683 % relative to the control treatment (A_0F_0). Results showed that application of purely wood ash, or in combination with a supplementary N and P source increased growth up to 8 months after trial establishment for wood ash applications up to 1200 kg/ha. This project demonstrated that 1200 kg/ha wood ash can safely be disposed of on a typical Zululand coastal sand with little environmental risk and no supressed growth, provided that it is balanced with an appropriate NP fertiliser.

Opsomming

‘n Veldproef met ses herhalings is in Oktober 2013 uitgelê met die doel om die uitwerking van verskillende vlakke hout as en kunsmis toedienings op die grond-voedingstof status, boom-voedingstof status en die groei-tempo van ‘n *Eucalyptus grandis x urophylla* hibried plantasie te bestudeer. Hout as by pulpmeulens word tans weggegooi op stortingsterreine. Toenemende onkostes vir storting en die omgewingsrisiko’s gebonde aan stortingsterreine, dryf maatskappye om verbeterde en meer omgewingsvriendelike metodes te ondersoek om van die as ontslae te raak. Hout as bestaan uit ‘n reeks karbonate, hidroksiede en kalsium bevattende minerale en is verantwoordelik vir die bekalkingseffek op die grond na toediening. Die veldproef is geleë naby Richardsbaai op ‘n sanderige grond met n lae bufferkapasiteit en pH van ongeveer 5.5. Die proef het vier hout as vlakke getoets, gekombineer met drie vlakke van bemesting: geen, 150g konvensionele landbou kunsmis (CV) óf 320g beheerd-vrystellende kunmis (CRF). Die kunsmismengsels en vlakke van bemesting is gebaseer op bestaande of voltooide bemestingseksperimente in die streek. Hout as vlakke was bereken in gekontroleerde laboratorium toestande en gebaseer op ‘n inkubasie studie met grond monsters verkry vanaf Richardsbaai, waarby toenemende vlakke suiwer landboukalk gevoeg is. Die veldproef het hout as vlakke van 0, 300, 600 en 1200 kg/ha getoets. Veld metings is op 4 en 8 maande na behandeling geneem. Die primêre doelwit van die studie was om te bepaal watter vlak hout as en kunmis kombinasie toegedien kan word, sonder om die grond-voedingstof status negatief te beïnvloed of ‘n potensiële skadelike uitwerking op die plantasie groei te veroorsaak. Die uiteinde van die studie was om die haalbaarheid van hout as toedienings op plantasie gronde te bestudeer relatief tot die huidige praktyk van storting, insluitend die risiko van moontlike skadelike newe-effekte.

Grondvoedingstatus was nie beduidend beïnvloed deur individuele hout as toevoegings nie, maar was eerder ‘n funksie van die tydsduur sedert behandeling. Grond koolstof, P anione, K^+ en Mg^{2+} konsentrasies het beduidend afgeneem in die periode van 4 - 8 maande na behandling. Die Ca^{2+} konsentrasies het toegeneem tussen 4 en 8 maande en terselfdertyd het Na^+ en B konsentrasies afgeneem. Die swaarmetaal status, spesifiek vir Cd, Hg, Cr en Pb, vir toevoegings van 0-1200 kg/ha

hout as was beduidend laer as toelaatbare vlakke in gronde op albei tydsintervalle. Die hout as het ‘n tydelike toename in grond pH veroorsaak. Die gemiddelde pH het tussen 0 - 4 maande toegeneem met 0.6 eenhede en gedurende 4 - 8 maande afgeem met 0.4 eenhede.

Blaarontledings en voedingstof assesseringsmetodes het sub-optimale konsentrasies vir P, K en Zn getoon op die ouderdom van 4 en 8 maande. Voedingstof konsentrasies is as sub-optimaal geklassifiseer, omdat konsentrasies nooit laer as kritieke waardes vir gebreksimptome was nie. Die inhoud van Cd, Hg, Cr en Pb in blare was aansienlik kleiner as 1 mg/kg op albei tydsintervalle. Die merkwaardige lae konsentrasies wat in die projek aangeteken is, word toegekryf aan die lae bio-beskikbaarheid van al vier elemente as gevolg van die edafiese faktore eie aan die Richardsbaai omgewing (en ook aan groot dele van die Zoeloelandse kusvlakte). Die groeireaksie (bepaal as biomassa indeks op ouderdom 8 maande) het gewissel van 13 % - 683 % groter as die kontrole behandeling (A_0F_0). Resultate het bewys dat toedienings van suiwer hout as, of hout as gekombineer met ‘n addisionele N en P kunsmisbron die groei postief beïnvloed tot op die ouderdom van 8 maande. Die studie het bewys dat 1200 kg/ha hout as veilig toegedien kan word op die sandgronde van die kusgebiede in Zululand, met minimale omgewingsrisiko en geen tekens onderdrukte groei nie, mits dit gebalanseer word met ‘n geskikte NP kunsmisbron.

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List of abbreviations

Al	Aluminium
ANOVA	Analysis of variance
B	Boron
BI	Biomass index
C	Carbon
Ca	Calcium
CCE	Calcium carbonate equivalent
Cd	Cadmium
CEC	Cation exchange capacity
Cr	Chromium
CRF	Controlled release fertiliser
Cu	Copper
CVF	Conventional fertiliser
DBH	Diameter at breast height
ECEC	Effective Cation Exchange Capacity
Euc	Eucalyptus
Fe	Iron
Hg	Mercury
ICP-OES	Inductively coupled plasma emission spectroscopy
K	Potassium
KZN	Kwazulu-Natal
LBC	Lime buffer capacity

MC	Moisture content
Mg	Magnesium
Mn	Manganese
Mo	Molybdenum
N	Nitrogen
Na	Sodium
NG	Natalgroup
P	Phosphorous
PAMSA	Paper Manufacturers Association of South Africa
Pb	Lead
PMB	Pietermaritzburg
Ppm	Parts per million
SE	South East
WA	Wood ash
Zn	Zinc

1. Introduction

1.1 Background and Justification

The South African industrial forest sector largely consists of plantation forestry and essentially relies on sufficient planning and best management practices for success. Plantation forestry is driven by production and primarily endeavours to maintain productivity for exotic species such as pine and eucalypt without compromising timber quality. The industry has to remain economically competitive and viable within the criteria set by market and industry forces. Exotic eucalypt species are favoured due to properties such as enhanced growth, improved water and light use efficiency and favourable wood properties (Stape et al., 2004). The increasing need for land and the difficulty of acquiring/re-acquiring afforestation licences has limited and reduced afforestation. This has compelled forest companies to invest and pursue new ideas to improve productivity, be more sustainable and meet the demand of mills at the same time. The majority of commercial plantations are used for pulp and paper production and mills are driven to be productive, profitable and sustainable at the same time. As with most major industries, processing of raw material generates unwanted waste by-products. In order for companies to gain entry to specific markets and business opportunities, it is important that these by-products are disposed of safely and within the guidelines and standards set by legislation and governing entities.

The limitations imposed on both the forest and the paper industries have opened a research gap for further investigation. A large portion of the energy required by pulp mills is commonly generated from mill residue materials such as wood and bark biomass. The fact that ash residues from this process contains large quantities of tree nutrients (Demeyer et al. 2001) opens up the possibility of re-introducing wood ash produced from mill energy plants to forest soils. The development of a practical and scientifically based plan for successful application of wood ash produced from paper mills on plantation soils, without compromising the efficiency and profitability of both sectors and more importantly remaining within environmental regulations, can be a major stepping stone for the South African forestry industry.

1.2 Wood ash use in forestry

The application of pulp and paper mill generated wood ash on plantation and natural forest soils is a concept that has been studied intensively for the last few decades. The concept of re-introducing processed of waste plant material removed from plantation and forest soils to offset nutrient losses from harvesting has greatly directed attention to this notion. The potential benefits from wood ash application to soil chemical and physical properties and tree growth should be balanced against the environmental risks imposed by its application. Studies have indicated wood ash has the potential to partly substitute nutrients removed from harvesting and greatly increase tree growth. In contrast some research has indicated possible risks of heavy metal contamination, potential water contamination, immobilisation and volatilisation of essential macro- and micro-nutrients and possible nutrient imbalances. Literature revealed one of the most influential variables governing the effects of wood ash on plantation soils and tree growth, to be the site conditions where the wood ash is applied to. For instance, Goodwin and Burrow (2006) stated the effect of wood ash on soil physical properties is significantly greater on sandy soils. The effects of wood ash and variability of results are governed by site conditions, soil properties, wood ash properties and abiotic factors (Demeyer et al., 2001; Aronsson and Ekelund, 2004; Pitman, 2006).

Wood ash is comprised of most essential macro and micronutrients and trace elements, but nutrient availability is governed by initial soil pH and pH increases ensuing from wood ash application (Patterson, 2001; Mandre et al., 2004; Schwenke et al., 2012). Wood ash can potentially replace several nutrients removed from whole tree harvest systems in a rotation, with the notable exception of nitrogen (Pitman, 2006; László et al., 2009). Wood ash amendments to forest soils have been done for a range of conditions in both the Northern and Southern hemispheres of the world, but this practice has not been implemented in South Africa. Literature revealed contrasting results regarding the effect of wood ash on tree growth; however Guerrini et al. (2000) reported growth increases in the range of 38-64% on three sandy soil types after wood ash and pulp and paper sludge applications in *Eucalyptus grandis* stands relative to chemical fertiliser. After 5 years increases of 75% were found in the Oxisol. Reduced height growths, induced by wood ash applications on nutrient deficient sandy soils, in *Picea abies* stands have been reported by Mandre et al.

(2004). Authors acknowledge the presence of heavy metals like Cadmium, Lead, Mercury and Arsenic in wood ash, but contrasting results are found throughout literature regarding their reactivity and elemental concentrations. Patterson (2001) stated the presence of Cd in wood ash can have negative effects due to bioaccumulation in plant and animal tissue. Site responses to wood ash amendments in South African conditions are undocumented, thus it is crucial these responses are documented and well understood before wood ash application is implemented on highly variable soil and site types in South African soils.

1.3 Problem statement and study objectives

Commercial pulp and paper mills produce large quantities of wood ash. The increasing accumulation and disposal of wood ash is a challenging task for mills and stringent environmental policies accompanied by increasing costs associated with disposing wood ash has driven the need for investigating alternative disposal methods (Demeyer et al., 2001). The potential of re-introducing nutrients produced from the combustion of biomass back into plantation soils fuels the notion of pursuing alternative disposal methods such as wood ash application to plantation soils.

The overview revealed several gaps in existing literature such as: (a) the effects of wood ash amendments on sites containing wood ash produced from site preparation (e.g. burning slash and stumps), (b) depth of downward transmission of wood ash in soil following application and its reaction in lower horizons, (c) the vagueness related to the legality of applying wood ash as a fertiliser in South Africa according to environmental legislation and (d) the reactivity of heavy metals in a sandy soil following application. This project will attempt to objectively investigate and gain a comprehensive understanding of the listed knowledge gaps, with the focal points on the Richards Bay pulp mill and the site conditions of surrounding plantations on the Zululand coastal plain.

The effect of wood ash application on the eucalypt plantations of the Zululand coast, KZN, has not been documented. The edaphic and climatic conditions given by this region is unique and no scientific evidence exists for effects of the application of wood ash produced from paper mills. This study will address the effects of wood ash on soil chemical and physical properties after application of different selected wood

ash application rates on a *Eucalyptus grandis x urophylla* plantation's growth several months following establishment. It will additionally investigate the type and quantity of additional nutrients (fertiliser) required to address imbalances that may result from using wood ash as a soil amendment. Lastly an assessment of the environmental risk imposed by heavy metal contamination and availability for varying application rates is undertaken.

This project is the first step whereby pulp and paper mill generated wood ash can safely be disposed of on nearby plantation soils; effectively saving the mill considerable storage and disposal costs, environmental fees as well as partly countering the effect of nutrient depletion from intensive short rotation forestry. The study aims to test whether application of mill generated wood ash from Richards Bay mill on sandy plantation soils with low buffer capacity is a real and viable alternate disposal method to the current method.

Study objectives:

1. Investigate the feasibility of incorporating wood ash in *Eucalyptus* stands and to establish if this method can be introduced as an environmentally acceptable ash disposal method, and –
2. To investigate the effects of wood ash on soil chemistry and *Eucalyptus* plantation growth following application.

1.4 Study approach

The project is based on a quantitative design and is divided into 2 sections; the pilot study and subsequent field trial establishment. The pilot study involved the reaction of soil samples from the field trial site with increasing quantities of lime in order to determine the soil pH buffer capacity. The results of the pilot study were incorporated into the field trial design. Four wood ash application rates derived from the baseline application rate determined in the pilot study were used for trial establishment in combination with 3 fertiliser treatments (one representing the unfertilised control). The trial was designed as a factorial with two main factors: wood ash application (4 levels) and fertilisation (3 levels). Tree stand nutritional status and heavy metal content in the foliage and soil samples were monitored at two intervals following trial establishment.

1.5 Hypothesis and research questions

Two hypotheses were formulated for the project; the first and most important hypothesis concerns the environmental risk of the disposal method under investigation and the secondary hypothesis the effect of wood ash on *Eucalyptus grandis x urophylla* growth.

A. H_0: The application of wood ash on plantation soils is a suitable alternative ash disposal method.
   H_1: Wood ash application on plantation soils is not a safe alternative ash disposal method and could impose environmental harm.

B. H_0: Wood ash application on plantation soils result in a positive growth response to *Eucalyptus* plantation species; increased growth and nutrient availability.

   H_1: Wood ash application on plantation soils result in suppressed *Eucalyptus* species growth; reduced growth and nutrient availability.

The key questions which are to be addressed in this study are:

1. Are there any changes in the exchangeable fractions of the base cations in the topsoil following ash application?
2. How does wood ash affect short term nutrient availability?
3. Does wood ash application affect foliar nutrient levels of eucalypts?
4. Does wood ash application (in the presence or absence of balanced NPK fertiliser supplements) result in improved or suppressed growth?
5. What is the concentration of heavy metal in wood ash and does it accumulate to hazardous levels in soils under tested levels of application?
6. What is the calcium carbonate equivalent of the wood ash and how will it affect the soil reaction once applied?

1.6 Limitations

Given the narrow timeframe for the project, project limitations are unavoidable. The project is limited to one commercial plantation species and soil type and restricts the

opportunity for extensive implementation if the project proves wood ash application is a viable disposal method. This is due to the Richards Bay pulp mill being the potential wood ash source if the project is implemented in the future, and as a result this determines the experimental conditions given by the immediate area. The monitoring of soil-water heavy metal content is hindered by the short project timeframe and geographic location of the project relative to the research institute, thus all conclusions for this notion is based on what is theoretically known. Monitoring of tree growth and soil chemical and physical changes is limited to several months for the purposes of this thesis and any long term changes that may be monitored in future fall outside the scope of this study.

Significantly lower wood ash application rates are studied in this project relative to literature reviewed. This is attributed to the soils that occur in the trial area and edaphic conditions ensuing from this. Low soil organic carbon content, high initial soil pH and low buffer capacities are features of the most common soil types in the area and significantly limit wood ash application rates. Lastly the project does not incorporate the economic viability of transporting wood ash from the mill to plantations for disposal if the disposal method is proven to be viable. This is excluded from the scope due to the narrow time frame of the project and the specific focus for this thesis.

1.7 Thesis structure

The thesis is comprised of a literature review (Chapter 2) that provides an in depth overview of what is currently known about the types of wood ash, under what conditions they are produced and their physical properties. The chapter furthermore provides an overview of the effects of wood ash on soil chemistry, tree growth, soil microbiology, soil-water and environmental challenges related to wood ash application. Chapter 3 provides a detailed description of the methodology used in the pilot study, experimental design and trial layout. Results for measurements and data collected at both intervals are described in Chapter 4, followed by the discussion (Chapter 5) and lastly the conclusions and recommendations chapter (Chapter 6).

2 Literature review

2.1 Introduction

Apart from Swaziland, South Africa is the sole producer of pulp and paper on the African continent, and annually produces close to 370 000 tons mechanical wood pulp, 1 500 000 tons chemical wood pulp, 316 000 tons newsprint, 970 000 tons printing and writing paper and more than 1 000 000 tons paper and paperboard (Mbendi, 2013).

The production of paper and pulp related materials produce by-products such as fly-ash, bottom ash and biomass ash from pulping processes and fuelling the boilers. Current disposal methods used for these materials in South Africa are dedicated landfills. Tightening environmental regulations and rising disposal costs have placed increased pressure on pulp and paper companies to further investigate alternative disposal methods (Demeyer et al., 2001). Wood ash can be used as a potential forest fertiliser if certain conditions are met (Goodwin and Burrow, 2006; Pitman, 2006; Kuokkanen et al., 2009). The implementation of whole tree harvest systems and planting of short rotation crops can likely lead to nutritional deficiencies in subsequent rotations and further removal of biomass in already N deficient sites can further aggravate the deficiency (Compton and Cole, 1991; Gonçalves et al., 2008). Intensive silvicultural management operations such as slash burning and shortened rotations can affect the ability of the site to supply a balanced set of nutrients to subsequent rotations. Burning of slash succeeding harvest operations result in increased short term nutrient availability and changes short term nutrient dynamics (du Toit and Dovey, 2005; Smith and du Toit, 2005; du Toit et al., 2008). Du Toit and Scholes (2002) showed considerable soil N, K and Ca nutrient losses resulting from wood harvesting and firewood collection; they additionally found that increased management intensity resulted in bigger nutrient losses and that in some instances nutritional stability can still me maintained depending on the site's soil buffering ability. The removal and burning of slash in *Eucalyptus grandis* stands show reduced N, P, K, Ca and Mg soil content relative to slash retained sites. In addition, burning of slash can partially retain P and base cations, but decreases are certain (du Toit, 2003). The author specified that N, K and Ca losses have to be managed to ensure nutritional sustainability. Du Toit (2003) showed variable degrees of P, K, Ca and Mg

losses in South African *Eucalyptus grandis* plantations ensuing from harvesting and slash burning operations. He additionally stated these losses could cumulatively add up to more substantial losses if rotations are not managed properly and thus lead to increased topsoil acidification. Similarly, recent findings by Titshall et al. (2013) identified harvest residue management, ground-based harvest mechanised harvest operations and to a smaller degree fertilisation as high disturbance activities. The authors recommend a more conservative approach be taken with regard to soil compaction from harvest equipment, and soil organic carbon and nutrient losses from intensive biomass and residue management practices to prevent any negative long term impacts on afforested sites. Titshall et al. (2013) concluded that South African soils appear to be resilient to forest management practices, but that there are a range of factors that need to be considered to maintain site nutrition sustainably.

Wood ash induces a strong alkaline reaction (liming effect) when used as a soil ameliorant and has the ability to correct nutrient deficiencies induced by base cation leaching and soil acidification (Demeyer et al., 2001; Ozolinčius et al., 2007). The effect of wood ash is effected by soil properties and application rate, it can potentially replace almost all nutrients (with the exception of N) removed per rotation from whole tree harvesting and other agricultural activities (Pitman, 2006; László et al., 2009). In addition, wood ash contains no sulphur and though not classified as a nutrient, it does not contribute to the carbon supply. The ability to safely dispose wood ash on plantations can potentially offset soil acidification, reduce potential nutrient deficiencies and sustain long term forest productivity (Jacobson, 2003).

This review focuses on the effect of wood ash on soil physical, chemical and biological properties, tree growth and environmental risks associated with the application thereof. It additionally incorporates the applicability of wood ash application to South Africa by investigating how South African legislation defines wood ash and the applicability of wood ash given South African soil conditions and the forestry industry.

2.2 Wood ash

2.2.1 Sources, types and toxic elements

Physical and chemical characteristics of wood ash produced from pulp and paper mills are variable. The variability and ash properties are determined by several factors; the type of material burnt, the plant parts making up the material (stem, leaves and bark), incorporation of other fuel sources, site conditions (soil and climate) and combustion conditions (Demeyer et al., 2001). Wood ash produced from the combustion of root and branch wood is richer in elements relative to ash produced from stem wood due to the inclusion of soil mineral components. Concentrations of elements produced from the combustion of bark and leaves have concentrations 5 to 10 times greater than that of stem wood (Pitman, 2006; Ozolinčius et al., 2007). Wood ash derived from bark can have high Ca and Si concentrations and ash produced from the combustion of mixed stem wood may contain high Mn, Ca and Si concentrations (Pitman, 2006).

Two types of wood ash are produced in mills and power plants using woody biomass as fuel; fly ash and bottom ash. Both ash types are distinguished by their reactivity, texture, heavy metal content and region of accumulation (Santalla et al., 2011). Fly ash is light-grey and accumulates or is captured in boiler emissions. It contains greater nutrient concentrations and increased reactivity; nutrients (K, Ca and Mg) are more easily available and rapidly released following application. Bottom ash, or also known as boiler ash, is less reactive, contains lower nutrient concentrations and lower heavy metal concentrations. The application of bottom ash ensures a long term and gradual release of nutrients and significantly reduces the risk of heavy metal contamination and is the recommended wood ash type to be utilised as a soil amendment (Pitman, 2006; Cassidy and Ashton, 2007; Santalla et al., 2011). Fly ash can contain possible high levels of damaging toxins and heavy metals, but it can be used as a soil ameliorant if leaching potentials of toxic elements are known (Kuokannen et al., 2009). In contrast, a review by Pitman (2006) on the use of wood ash in forestry, emphasized fly ash contains increased cadmium, chromium, copper, lead and arsenic concentrations and is not suitable for application. Refer to Table 2.1 in section 2.2.3 for individual mean and range heavy metal concentration values found in wood ash produced from different sources. In addition, Table 2.1 shows the

heavy metal concentration differences for wood ash and fly ash relative to other wood ash sources.

Wood ash may contain several dioxins and furans (Elliot and Mahmood, 2006; Matysik et al., 2001; Pitman, 2006) including harmful organic compounds such as polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans (Matysik et al., 2001). Dioxins and furans are formed as a product of burning salt laden woody material and the presence of chloride determines the concentration of chlorinated organics in ash (Elliot and Mahmood, 2006). Variable dioxin and furan concentrations are additionally determined by boiler operating conditions, temperatures and fuel salt content (Demeyer et al., 2001; Elliot and Mahmood, 2006). The application of wood ash at rates exceeding 10 t ha^{-1} can lead to possible toxicity, but contamination from radionuclides, heavy metals and dioxins in wood ash is insignificant and would likely not affect ecosystem function (Pitman, 2006). Pitman (2006) additionally found that leaching of dioxins and furans from wood ash is improbable, due to the absorbent behaviour of both compounds. This suggests that both compounds persist in the soil if perhaps incorporated into the soil by means of wood ash applications.

Wood ash is largely applied in three usable forms; loose, crushed and granulated. All three ash forms have differing chemical compositions; a higher grade of processing yields lower Ca and increased P concentrations (Pitman, 2006). The risk of heavy metal contamination, N-leaching, root damage and possible harm to soil biota and vegetation is greatest in loose form and high application rates (Jacobson, 2003). Loose wood ash releases Ca, K and Na more rapidly than granulated or crushed wood ash (Pitman, 2006). The reactivity and potential damage can be reduced by stabilising the product, effectively changing the product from a loose to crushed or granulated texture. A granulated form is achieved by intermixing loose ash with water and pressing it into balls ranging 4 mm – 20 mm in size, subsequently the product is dried to a moisture content of less than 5% (Kellner and Weibull, 1998). In Finland a crushed or granulated form is achieved mixing water and ash to the desired MC in a mixer and feeding the mixture into a drum granulator, subsequently the product is screened and crushed to the required granule size (Väätäinen et al., 2001). It is essential to factor in particle size during implementation if wood ash is to

be used as a potential soil ameliorant; small particle sizes and powdery textured ash could easily result in losses during transportation and application in windy conditions. The granulation methods described by Kellner and Weibull (1998) and Väätäinen et al. (2001) can effectively be used to adjust application rates to safer quantities (Pitman, 2006), and reduce potential harm to soil biota and vegetation stemming from potential over-application (Jacobson, 2003).

2.2.2 Physical attributes

Wood ash particle size is variable and largely depends on the degree of combustion. Etiégni and Campbell (1991a) studied the physical and chemical properties of wood ash and the effect of combustion temperature. The authors recorded an average particle size of 230 µm, using a statistical plot of ash particle size distribution for powdered ash. They attributed large particle sizes to large porous carbon particles produced during incomplete or partial combustion. Ribeiro et al. (2010) measured particle sizes in the range of 0.040 to 2 000 µm for wood ash produced in a pulp mill boiler. The particle size distribution measured by the authors was expressed as the mean equivalent diameter of a spherical particle.

Wood ash is essentially hydrophilic and swelling occurs when mixed with water; thus possessing the ability to absorb and retain water. This property allows for the potential effect of increased water-holding capacity and enhanced nutrient availability as nutrients are taken up in solution (Etiégni et al., 1991a; Goodwin and Burrow, 2006; Pitman, 2006; Santalla et al. 2011).

2.2.3 Chemical attributes

2.2.3.1 Macro- and micronutrients

The effects of combustion temperature on the chemical properties of wood ash are well documented. Etiégni and Campbell (1991) studied the effects of combustion temperature on chemical content and wood ash yield. They noted ash yield decreases significantly with increased combustion temperatures; ash yield decreased by nearly 45% with temperature increases from 538 – 1093 °C. The extent of nutrients released by the combustion process is related to the temperature. The maximum amount of nutrients is released between 600 - 900 °C, with the

highest concentrations of macronutrients released at 500 - 800 °C (Etiegni et al. 1991a). The K, Na, Zn and carbon content decreases with increased temperatures and metal ions increase or remain constant (Etiégni et al. 1991a; Pitman, 2006). In boiler ash, K and S only become volatilized at temperatures ranging from 800 - 900 °C and 1000 - 1200 °C, respectively. K losses amounted to 63% and 90% and S losses ranged 7 - 55% (Naylor and Schmidt, 1986). Carbonates and bicarbonates become more prevalent at a combustion temperature below 500 °C and oxides above 1000 °C in industrial wood-fuelled boilers (Etiégni et al., 1991a).

Wood ash contains variable macronutrient concentrations. The quantities of C and N in wood ash are usually low and at times completely negligible; this is attributed to the oxidation of both elements at combustion (Demeyer et al., 2001). Potassium from wood ash is extremely soluble and is easily leached by water (Etiégni et al., 1991a and Ulery at el., 1993). In pelleted form P remains soluble and can improve soil fertility (Kuokannen et al., 2009). Microelement concentrations are just as variable as macro-elements (Demeyer et al. 2001). Micronutrients are found in optimum concentrations in wood ash and can be used as a potential micronutrient-fertiliser (László et al., 2009 and Mandre, 2006). The Ca and Mg contents of wood ash are lower relative to current fertiliser agents used in agriculture and Fe is the most abundant microelement (Demeyer et al., 2001).

A detailed breakdown of several wood ash sources is presented in Table 2.1. In addition, elemental concentrations for bottom and fly ash are shown in Table 2.1, taken from a report by Alakangas (2005) on the properties of wood fuels in Finland. Values reported by Etiegni et al. (1991a), Huang et al. (1992), Ohno and Ehrich (1993) and Muse and Mitchel (1995) represent total concentrations for individual elements. The values had been adapted from Demeyer et al. (2001), Alakangas (2005) and Siddique (2008).

Table 2.1: Elemental concentrations of several wood ash sources. Adapted from Demeyer et al. (2001), Siddique (2008) and Alakangas (2005).

Source	Ashes of wood and bark (mg kg^{-1})		Ashes of paper and pulp (mg kg^{-1})		Wood Material (mg kg^{-1})	Wood Material (mg kg^{-1})	
						Bottom ash	Fly ash
Element	Etiegni et al. (1991 a)	Huang et al. (1992)	Ohno and Ehrich (1993)	Muse and Mitchel (1995)	Siddique (2008)	Alakangas (2005)	
Organic Carbon				247 000	-	-	-
N	600	900		4520	-	-	-
P	14 000	6 900	1 800	3 000	-	-	-
K	41 300	28 600	10 300	13 300	-	-	-
Ca	317 400	109 400	94 900	120 000	-	-	-
Mg	22 500	16 200	6500	7 730	-	-	-
S	4 455	6 800			-	-	-
Na	3 400	1 600	6 700	1 410	-	-	-
Fe	19 500	3 300	14 300	6 260	5 900-6 100	-	-
Zn	700	794	423	183	380-420	15-1 000	40-700
Mn	6 693	3 470	3 300	2 600	2 440-2 750	2 500-5 500	6 000-9 000
Cu	145	78	151	67	41-46	15-300	200
Al	23 650	1 300	82 100	12 500	4 000-4 500	-	-
Mo	114		61	15	5.6-6.7	-	-
B	8	127		95		-	-
Pb	130	66	32	72	29-35	15-60	40-1 000
Ni	47	12	65	16	6-8	40-250	20-100
Cr	86	14	1036	75	12-14	60	40-250
Co		4		14		0-7	3-200
Cd	21	3	<1	2	5.5-6.1	0.4-0.7	6-40
Ba			549	588	220-300	-	-
As					42-53	0.2-0.3	1-60
Hg					0.05-0.08	0-0.4	0-1
Se					0.53-0.64	-	5-15
V						10-120	20-30
Ag					0.2-0.4	-	-

2.2.3.2 Liming and potential other attributes

As stated earlier; arrays of conditions affect the physical and chemical properties of wood ash and attribute to the unique composition of wood ash. Wood ash has a high acid neutralising capacity and induces a liming effect (Etiégni et al., 1991a; Demeyer et al., 2001; Pitman, 2006; Kuokannen et al., 2009; László et al., 2009). The degree of combustion and temperature greatly affects the elemental concentrations in wood ash (Etiégni et al., 1991a). A significant proportion of the chemical composition of wood ash comprises of a mixture of oxides, silicates, hydroxides and carbonates and the solubility of some of these oxides and hydroxides induce the liming effect from wood ash application (Mandre, 2006). Etiégni and Campbell (1991a) recognised two neutralising points; the first major point being hydroxides and secondly carbonates. Table 2.2 presents a detailed illustration of wood ash elemental concentrations relative to limestone.

Table 2.2: The mean and range of elemental concentrations for industrial wood ash relative to ground limestone (Risse and Harris, 2011).

Element	Wood Ash*		Limestone
	Concentration (%)	Range (%)	Concentration (%)
N	0.15	0.02 - 0.77	0.01
P	0.53	0.10 - 1.40	0.06
K	2.60	0.10 – 13.0	0.13
Ca	15	2.50 – 33.0	31.0
Mg	1	0.10 - 2.50	5.10
S	-	-	-
Na	0.19	0.00 - 0.54	0.07
Fe	0.84	0.20 - 2.10	0.29
Zn	233	35.0 - 1 250	113.0
Mn	0.41	0.00 - 1.30	0.05
Cu	70	37.0 – 207.0	10.0
Al	1.60	0.50 - 3.20	0.25

Element	Wood Ash*		Limestone
	Concentration (%)	Range (%)	Concentration (%)
Mo	19	0.00 – 123	-
B	123	14.0 – 290.0	-
Pb	65	16.0 – 137.0	55.0
Ni	20	0.00 – 63.0	20.0
Cr	57	7.00 – 368.0	6.0
Co	-	-	-
Cd	3	0.20 – 26.0	0.70
Ba	-	-	-
As	6	3.00 – 10.0	-
Hg	1.90	0.00 – 5.0	-
Se	0.90	0.00 – 11.0	-
V	-	-	-
Ag	-	-	-
Other Chemical Properties			
$CaCO_3$	43% (22-92%)		100%
pH	10.4 (9-13.5)		9.9
% Total solids	75 (31-100)		100
*Mean (and range) taken from analysis of 37 ash samples			

2.3 Nutritional sustainability in South African soils

2.3.1 Soil nutrition

Wood ash application as organic matter can greatly improve physical properties of forest soils, especially on sandy sites (Goodwin and Burrow, 2006). The effects of applying wood ash as a soil ameliorant is predominantly governed by application rate and soil type (Pitman, 2006). It is essential to gain a comprehensive understanding of how South African forest soils react to different plantation forestry practices, as wood ash applications could potentially alter soil chemical and physical properties.

This section describes the impact of different slash management regimes for a widespread soil and climatic conditions in South Africa.

The South African plantation forestry industry can be divided into three major forestry economic areas; South East Mpumalanga, Eastern Mpumalanga and the Kwazulu-Natal Midlands (Smith et al., 2005). Four major soil forms are found within these regions; Ecca sandstone, Highveld granite, Natalgroup (NG) sandstone and Pietermaritzburg (PMB) shale. SE Mpumalanga's forestry economic region is dominated by Highveld granite and Eastern Mpumalanga by Transvaal shale and Lowveld granitic soils. The KZN Midlands are mainly dominated by NG sandstone and PMB shale soils. A forth zone that is of economic importance, due to the rapid growth rate, is the Zululand coastal plain. This zone is dominated by recent deposits of arenosols that have very low soil carbon contents, and very small nutrient pools in soils and litter layers (du Toit et al. 2014).

Within an ecosystem nutrients occur in nutrient pools; nutrient pools comprise of above and belowground biomass, the forest floor and the soil. Pools are divided into 2 categories, namely readily available and potentially available pools. Major inputs to these pools are: atmospheric deposition, N-fixation, fertilisation and weathering. Processes resulting in loss of nutrients form these pools are: erosion, weathering, harvesting, volatilisation and fire (du Toit et al., 2014). Nutritional sustainability of plantation soils largely depends on the size of the bio-available pool (du Toit and Scholes, 2002). Du Toit and Scholes (2002) furthermore indicated that larger nutrient pools increase the buffering ability of soil against short term nutrient changes induced by forest management practices. Smaller nutrient pools have a limited buffering ability and result in larger variations in system stability following disturbances. Du Toit et al. (2014) furthermore stated that slash removal does not necessarily induce soil acidification and severe reductions in base cation concentrations; the minor reductions can easily be offset using fertilisation. However reduced stand productivity has been reported on sensitive soils, thus emphasizing the importance of short term nutrient pools from residues as a nutrient source on plantations. Du Toit and Scholes (2002) concluded that the degree of site disturbance is largely determined by the intensity of nutrient removal and site fertility.

The process of estimating nutrient flux changes induced by various biomass removal intensities and management regimes is best described by the pINS index proposed by du Toit and Scholes (2002) for South African conditions. Table 2.2 presents a summary of the net nutrient flux estimations for three harvest intensities for eucalypt pulpwood sites from the coastal regions of Zululand (Zld), KZN midlands (Mid) and the Mpumalanga escarpment (Mpu), as published in Ackerman et al. (2013); du Toit et al. 2014. Scenario A represented a regular harvest operation with 75% stem wood removal. Scenario B was similar to A, but integrated slash burning. Scenario C represented whole tree harvest; 75% of the total above-ground biomass was removed in addition. Negative values signified net losses for the system and losses were significant for scenario B and C relative to A. Net flux estimations, shown in Table 2.2, did not incorporate soil nutrient pools as shown in the pINS indices in Table 2.3.

Table 2.3: Eucalypt net nutrient fluxes (kg ha^{-1} a^{-1}) in three regions for three common harvesting intensities. Scenario A represented a regular harvest operation with 75% stem wood removal. Scenario B was similar to A, but integrated slash burning. Scenario C represented whole tree harvest; 75% of the total above-ground biomass was removed in addition Adapted from Ackerman et al. 2013.

	Scenario A					Scenario B					Scenario C				
Region	N	P	K	Ca	Mg	N	P	K	Ca	Mg	N	P	K	Ca	Mg
Zld	19	2	-1	-9	11	-19	0	-13	-39	5	-13	-1	-24	-43	0
Mid	8	2	-2	-4	-5	-34	0	-17	-33	-12	-30	-2	-30	-46	-19
Mpu	18	1	-14	-25	-8	-79	-3	-41	-60	-25	-41	-4	-58	-89	-29

As stated previously the pINS indices proposed by du Toit and Scholes (2002) incorporated readily and potentially available pools and harvest intensity. The indices presented in Table 2.3, adapted from du Toit et al. (2014), were calculated only for

readily available nutrient pools due to a data shortage for potentially available pools. Values of 1.0 represented a theoretical threshold for sustainability and meant that net nutrient losses were more than 1/10 of the readily available nutrient pool and negligent management could lead to soil nutrient depletion after several rotations. Values of 2.0 and 3.0 represented losses of 1/100 and 1/1000 of the readily available nutrient pool and are thus not viewed as a threat to nutritional sustainability.

Table 2.4: pINS indices for eucalypt pulpwood sites on the main forest economic regions of SA for two harvest intensities and varying soil conditions, adapted from Ackerman et al. (2013) and du Toit et al. (2014).

		Scenario A					Scenario C				
Region	Lithology	N	P	K	Ca	Mg	N	P	K	Ca	Mg
Zld	Arenite	net gain	net gain	1.6	1.7	Net gain	1.3	2.0	1.0	1.2	1.5
Zld	Sedimentary	net gain	net gain	1.7	1.8	net gain	1.2	1.3	1.0	1.2	1.6
Mid	Dolerite	net gain	net gain	2.8	2.6	2.1	1.4	1.8	1.6	1.6	1.5
Mid	Shale	net gain	net gain	2.1	2.0	1.9	1.5	1.6	1.2	1.1	1.3
Mpu	Granite	net gain	net gain	1.8	1.7	1.9	1.5	1.5	1.2	1.1	1.4
Mpu	Gneiss	net gain	net gain	1.9	1.7	1.6	1.3	1.6	1.3	1.2	1.2

The net flux estimations for scenario A, showed in Table 2.3, indicated net gains for N and P in all regions, and as a result pINS indices were not determined for both elements. The moderate losses for K, Mg and Ca, shown in Table 2.3, were

furthermore expressed as pINS indices, shown Table 2.4, for each element in scenario A. The losses for K, Ca and Mg were moderate and did not impose a severe threat to nutritional stability for the harvest intensity outlined in scenario A. However, in scenario C, pINS indices for N decreased to levels very close to 1.0 and indicated a potential loss of site productivity after a number of rotations. Indices for K, Ca and Mg showed losses for all nutrients relative to scenario A, but index values remained slightly above 1.0 and showed that the soils were moderately buffered against the depletion of K, Ca and Mg. Results showed that similar to N, after several rotations the soil system might run the risk of nutrient depletion and reduced productivity. Indices for P showed lesser losses for all regions, but as stated earlier P is easily and economically substituted with fertiliser.

Full tree harvest systems can potentially induce nutrient deficiencies due to slash removal (above-ground biomass). South African literature verifies the risk of reduced site productivity and nutrient depletion if sound management practices are neglected or additional nutrients are not supplied to the soil. Full tree harvest systems (slash removal) coupled with slash burning could furthermore increase nutritional losses to soil bodies. The effect of slash management and subsequent wood ash production on plantation soils is described in the following sections.

2.3.2 Arenosols

This section investigates arenosol characteristics and chemical properties. Arenosols have a sandy texture and comprise of little organic matter (Ngole, 2010; Ngole and Ekosse, 2008). South African and Namibian arenosol topsoils have variable pH values; soils can range from acidic to basic (Hartemink and Huting, 2008). Hartemink and Huting (2008) studied the land cover, extent and characteristics of arenosols in Southern Africa. Data gathered revealed South African arenosols typically have low organic carbon content and that the organic carbon rarely exceeds 10g kg^{-1} and soil N content is largely below 0.7g kg^{-1}.

Ngole and Ekosse (2008) studied the physico-chemistry and mineralogy related to productivity of an arenosol, luvisol and vertisol. Arenosol samples identified quarts as the dominant mineral in this soil type and amounts of kaolinite and feldspars; the soil comprised primarily of sand and had the smallest clay percentage relative to other

soils in the study. Characteristics identified for the considered arenosol: It comprised of a sandy structure and contributed to the lowest water-holding capacity, moisture content, organic matter content and CEC values observed for the arenosol relative to other soils. Some known effects of wood ash application on sandy soils are described by Mandre et al. (2004) and Guerrini et al. (2000). Experimental conditions and results are described in Section 2.4.

Literature showed that wood ash can potentially raise the soil pH and is considered an organic waste or by-product given pure or uncontaminated wood is used (Section 2.10). In arenosols, increases in soil pH and organic carbon content can lead to increases in CEC (Hartemin and Huting, 2008). The resilience of a plantation soil system largely depends on the net nutrient flux relative to the size of the bio-available pool (du Toit and Scholes, 2002). This means small nutrient pools (characteristic of sandy soils) will show a larger variation in soil system stability relative to larger nutrient pools, brought on by various forest management operations.

Hartemin and Huting (2008) concluded that Southern Africa has 6.5million Ha poor and nutrient deficient arenosols and supplementary nutrient sources are needed to improve agricultural productivity. This furthermore enforces the need for exploration of additional soil nutritional inputs such as wood ash and products of biomass combustion.

2.3.3 Slash burning and ashbeds

As there is currently no research on the application of wood ash on South African plantation forestry soils, it could be argued that the soil chemical and physical changes resultant from wood ash produced from slash burning could represent potential soil reactions from boiler produced wood ash. It is important to recognize that wood ash produced from slash burning includes a heat component and the chemical composition likely differs from boiler produced wood ash. This section reports on South African and international findings regarding the effect of slash burning on forest soils.

Forests are dynamic systems whereby nutrients are recycled continuously. Simply nutrients are taken up by roots from the soil, internally translocated within the tree, recycled back into the soil as litterfall, fine root turnover and crown leaching and lastly decomposed on the forest floor (Laclau et al., 2003). Total aboveground nutrient losses for slash-and-burn (clearing) of forests can be considered as the most severe type of known disturbances (Giardina et al., 2000). Slash burning is the process of reducing forest residues produced at harvesting and mobilising it to prepare the site for future establishment. It differs from wood ash application in that application is done with the intention of improving site nutrition and simultaneously disposing wood ash in a more environmentally sound way. Burning of forest residues can result in substantial nutrient losses stemming from erosion, burning, volatilisation and leaching (Hart, 1985; Gonçalves et al., 1995; Giardina et al., 2000; Kauffman et al., 2009)

An integral part of silvicultural operations in plantation forestry is residue management; bark, woody components and branches are broadcasted and burnt during site preparation. The burning of slash temporary releases vast amounts of nutrients and subsequently increases growth (Giardina et al., 2000; du Toit and Dovey, 2005; Gonçalves et al., 2008; du Toit et al., 2008). Gonçalves et al. (2008) studied the effects of various residue management practices on soil fertility and growth on *Eucalyptus grandis* plantations in Brazil. Results indicated significant, but temporary increases in tree productivity for slash retained and burnt sites (soil organic carbon fluctuations observed at 6, 10 and 26 months). Reduced productivities were recorded for sites stripped of all residues (bark, litter and slash). In addition, the findings confirmed that the initial soil fertility increase in the upper soil layers was not enough to offset substantial nutrient losses ensuing from burning and the negative effect on long term soil fertility. Losses measured by Gonçalves et al. (2008): 82 % Biomass, 86 % N, 60 % P, 49 % K, 11 % Ca, 29 % Mg and 84 % S. Losses were attributed to volatilisation and ash losses. Similar carbon losses from forest slash burning reported by Kauffman et al. (2009) ranged between 62 % - 80 %. Fire severity primarily determines soil nutrient concentration losses following slash burning (Kauffmann et al. 2009). Kauffman et al. (2009) quantified various levels of deforestation and slash fire severity on nutrient losses and redistribution in a Brazilian tropical dry forest. Results indicated that increased fire intensity reduced

C and N concentrations and increased Ca concentrations. Burning of residues can decrease soil microbial biomass (Giardina et al., 2000; Gonçalves et al., 1995; Mendham et al., 2002). Soil temperatures in excess of 127 °C can sterilize the soil and induce severe microbial biomass mortality (Giardina et al., 2000).

Mendham et al. (2008) tested a range of management operations on *Eucalyptus globulus* in South-Western Australia on two contrasting soils. One soil was a fertile, deeply weathered, red Ferralsol and the second a low fertility sandy soil. In the 0-10 cm soil layer, the Ferralsol had an organic carbon content of 49.8 g kg^{-1}. The sandy soil had an organic carbon content of 27.7 g kg^{-1}; both soils were fairly rich in organic C. After 10 years, residue management had no effect on soil C, total N and P, but exchangeable soil cation stores were greatly affected (fluctuations) in the 0-10 cm soil surface layer. Slash retention did however result in increased soil N mineralisation relative to burning in the first initial years. Plantation productivity varied greatly for both soil types; no responses were noted for residue management or nutrient additions on the Ferralsol. Strong responses were observed on the grey sandy site for slash retained and added N sites. Burning and slash retention increased soil Ca content. Residue burning increased soil pH in the red Ferralsol and increases on the sandy sites were recorded at a soil depth of 10-20 cm. Effects disappeared after approximately 1 year on both soils, but the Ferralsol maintained a higher soil pH compared to unburned treatments.

Gonçalves et al. (2008) studied the effects of different residue management operations on 7 year old *Eucalyptus grandis*. Soil was identified as a Haplic Ferralsol. Soil characteristics: loamy, dystrophic and with a soil C content of 15.2 g kg^{-1} in the 0-10 cm sub-surface layer. The soil was predominantly sand. Burning of slash resulted in large and gradual increases in exchangeable Ca and Mg. Decreases were noted in exchangeable Al; changes were recorded at a soil depth of 10 cm. Soil K content increased 0.8 years after slash burning and decreases were attributed to leaching. Slash burnt sites had the lowest N mineralisation rates and in addition the highest nutrient losses were observed for these sites. Nutrient losses were attributed to volatilisation, leaching and erosion. High volume growth was recorded on sites succeeding slash burning, but Gonçalves et al. (2008) specified that recurrent nutrient losses will greatly reduce the total soil nutrient pool and in

several rotations potentially have disastrous implications for productivity if not managed adequately.

Du Toit et al. (2008) observed the effects of different slash management treatments on soil chemical changes, soil water content, foliar nutrient levels and stand productivity on *Eucalyptus grandis* for a full rotation. Additional treatments included a topsoil disturbed and supplementary fertiliser added treatment. The experimental site was located in Karkloof (Kwazulu-Natal) on a dolerite and shale derived soil. The soil was rich in humus and had an organic C content of 70 g kg^{-1}. The soil was essentially a clayey soil. Slash burnt sites showed increased Ca, Mg and extractable P concentrations at 1 year after treatment. Slash burnt and fertilised sites showed increased macronutrient concentrations, leaf area development and volume growth. Burnt sites showed increased maximum organic carbon content and increases remained fixed over the rotation. Slash burnt treatments showed increases in topsoil pH at 0 – 2 after treatment implementation, and soil pH values reverted back to initial values after 7 years. Cation exchange capacity increased the most for burnt sites; this was attributed to the changes in exchangeable base cations and extractable acidity. The sum of exchangeable base cations increased by approximately 3 $cmol.kg^{-1}$ at year 1 and the effect declined by year 2. Major P increases were noted at year 1 on the burnt sites relative to other treatments.

Deleporte et al. (2008) studied the effects of slash and litter management on soil chemical properties and growth on eucalypts in the Congo. Second rotation eucalypt clonal hybrids with *Eucalyptus alba* as the mother tree was used in the experiment. The soil was described as a Ferralic arenosol and was essentially a homogenous sandy textured soil. The soil was acidic and had limited available nutrients, low exchangeable cation concentrations, organic matter content and cation exchange capacity. Burnt treatments were defined as stemwood removal by means of harvesting and the burning of all slash and litter. Soil nutrient concentrations did not vary considerably between burnt, biomass removed or retained treatments. The most important treatment effects: Soil analyses showed at 8 years after establishment slash burnt treatments had the highest concentrations of exchangeable Ca and Mg relative to slash removed, double slash applied and stemwood harvested treatments at a soil depth of 0 - 10 cm. The greatest height and

volume growth was recorded for burnt treatments at 11 months after trial establishment, but growth slowed down at 2 years of age relative to slash retained treatments.

The effects of slash/residue management on soil chemical and biological processes is well documented, however no literature was assimilated on the effect of wood ash application on existing ash beds.

2.4 Lime – soil mechanism

This section describes the mechanisms responsible for initiating the changes in soil chemistry ensuing from wood ash (liming material) application and includes the soil chemical changes noted in other international projects relative to the expected soil reactions. It additionally discusses the resultant growth responses from wood ash additions on forest soils.

2.4.1 Wood ash - soil mechanism

In order to fully understand why wood ash application or the presence of wood ash elicits the soil chemical responses described previously, the mechanism driving the soil changes must be understood.

The application of an organic material may affect the electrical charge systems in a soil. The movement of nutrients are greatly affected by permanent and variable-charges. Variable charge systems are produced as a result of the protonation and deprotonation of surface hydroxyl groups (Sollins et al., 1988). Soils can contain both permanent and variable charge surfaces, but are more likely to be occupied by a single one. Variable-charge soils are more frequently found in the tropical regions; tropical conditions accelerate the development of variable-charge minerals due to warmer and more humid soil conditions. Mineralogy greatly determines whether a soil is dominated by a permanent or variable-charge; oxisols, ultisols, histosols and andisols are primarily variable-charge soils (Sollins et al., 1988).

Variable-charge soils are electrically neutral when the pH is equal to the pH at the point of zero charge (PZC) (Sollins et al., 1988). Sollins et al. (1988) furthermore stated organic matter is a crucial regulatory material with regards to soil pH. Highly

weathered tropical soils tend to be acidic if copious amounts of organic matter are present in the soil, and soil organic matter and silicate clays are the only main components with a low pH at the point of zero charge. The addition of organic matter, in particular wood ash, will increase the soil pH and thus the variable charge of the soil. The buffering capacity of a soil is crucial in determining the rise in soil pH from lime application. Variable-charge soils are characteristically poorly buffered at low pH and well buffered at high pH values; variable-charge soils have the ability to take on an additional charge (Fox et al., 1981). If lime is applied to a highly weathered soil (e.g. tropical oxisol) the pH increases and subsequently the cation exchange capacity. Fox et al. (1981) furthermore specified that the additions of lime to variable-charge soils can result in reduced P availability due to increased Ca concentrations.

Variable-charge soils can become acidic in the matter of a very short time if the land use continuously removes basic cations from the soil pool and the soil pH decreases below the buffered zone (Fox et al., 1981).

As stated earlier, wood ash comprises of carbonates, hydroxides and oxides. The soil changes in soil chemistry, macro and micronutrient availability and concentrations, is best described to the similar mechanisms responsible for the soil chemical changes induced from liming a soil body with dolomitic or calcitic liming materials. The loss of Ca^{2+} and Mg^{2+}through leaching and addition of H^+ to soil body by decomposition of plant material / organic matter and nitrification of NH_4^+ added as a fertiliser to soils are the main reasons for soil acidification. Equation 1, a simplification of the acidification process, best describes the soil acidification (Crozier and Hardy, 2003).

Equation 1: $NH_4^+ + 1^1/_2\ O_2 \longrightarrow NO_3^- + 4H^+$

Acidification furthermore increases as a result of the reaction of clay minerals with the added H^+; the process releases Al^{3+}. The addition of lime, dolomitic lime (Mg) in this case, is illustrated in Equation 2. The lime dissolves in the soil to release hydroxides and bicarbonates into the soil solution, subsequently the hydroxides and

bicarbonates react with H^+ and Al^{3+} to produce a weak acid as shown in Equation 3 and 4 (Crozier and Hardy, 2003).

Equation 2: $CaMg(CO_3)_2 + 2H_2O \longrightarrow Ca^{2+} + Mg^{2+} + 2HCO_3^- + 2OH^-$

Equation 3: $Al^{3+} + 3OH^- \longrightarrow Al(OH)_3$

Equation 4: $H^+ + OH^- \longrightarrow H_2O$

The characteristic reaction of a calcitic lime is similar to dolomitic lime. The lime supplies the soil with Ca^{2+} and Mg^{2+} in the form of a carbonate, hydroxide or oxide ($CaCO_3$, CaO or CaO). These compounds dissolve in the soil solution and the CO_3^{2-}, OH^- or O^{2-} reacts with the H^+ to produce carbonic acid (H_2CO_3) or water. The removal of H^+ furthermore results in the reaction of free Al^{3+} with OH^- and the production of insoluble $AlOH_3$. Any hydrogen remaining in the clay fraction of the soil is released into the soil in an attempt to maintain chemical equilibrium as acidity is neutralised, and free Al^{3+} again forms an insoluble compound as shown in Equation 3. H^+ neutralisation is sustained until all carbonates, oxides and hydroxides are exhausted. Carbonic acid will mostly dissociate and form CO_2 or H_2O. Any excess H^+ is converted into water, and free Ca^{2+} and Mg^{2+} replaces the released H^+ and Al^+ on the exchange (Mullen et al., 2007).

The potential effects of wood ash on soil chemistry are best described by results gathered from international studies, described in the subsequent section.

2.4.2 International case studies and findings

The application of wood ash increases soil pH and decreases soil exchangeable acidity (Demeyer et al., 2001; Kuokannen et al., 2009; Mandre et al., 2004; Mandre, 2006; Perucci et al., 2008; Pitman, 2006; Saarsalmi et al., 2012). The application of wood ash can greatly improve physical properties of forest soils, especially on sandy sites (Goodwin and Burrow, 2006). Soil mineralogy, soil electrical charge and buffer capacity greatly affects the rise in soil pH ensuing from lime applications. Increases in soil Ca content can result in reduced P availability due to the resulting increase in soil pH (Fox et al., 1981). In addition, Elliot and Mahmood (2006) stated that the phosphorous availability of wood ash varies significantly and can range from 30% -

70%; availability is a product of soil form and chemical characteristics. The variable P availability of wood ash can additionally be attributed to the soil pH buffering capacity (Etiégni et al., 1991a). Etiégni et al. (1991a) concluded that P availability is at a maximum for a soil pH in the range of 6.0 - 7.0 and decreases over 8.0 pH units. The application of wood ash may increase plant available P and K, but the effect is brief (Ohno and Erich, 1994). Ohno and Erich (1994) studied the effects of wood ash on P and K availability and results showed increased plant available P and K for the first 25 weeks and a gradual decline thereafter.

The application of wood ash on nutrient poor sandy soils can lead to increased soil pH, increased K and Ca concentrations, decreased N and P concentrations and potential nutrient imbalances (Mandre et al., 2004). Mandre et al. (2004) studied the effects of 0.25, 0.5 and 1 kg m^{-2} wood ash applications on *Picea abies* (Norway spruce) at the age 4 years. Literature reviewed by Goodwin and Burrow (2006) stated that the alkalinity of wood ash could increase N volatilization as a result of soil pH increases from wood ash additions. This can be explained by research done by Schwenke et al. (2012). Schwenke et al. (2012) investigated the relationship for the degree of nitrogen volatilisation and the application of different N formulations (urea and ammonium sulphate) at different times of the year. Results showed that a rise in soil pH increased the loss of nitrogen if urea was used as an N source, along with several other contributing factors such as temperature, crop canopy and soil moisture content. The over-application of wood ash can potentially increase soil pH substantially and result in a high pH zone, thus increasing nitrogen volatilisation if urea based fertiliser is used.

The application of 11 Mg ha^{-1} wood ash on 67 year old *Pinus pinaster* stands in Mediterranean climatic conditions increased the leaching of base cations and P, and affected soil C and N dynamics (Gómez-Rey at al., 2013). Gómez-Rey et al. (2013) observed the effects of loose and pelleted wood ash applications on nutrient leaching, nutrient dynamics and fertility. Shortly after application increased leaching of base cations and P was observed and as a result increased soil nutrient availability and reduced soil acidity. This was more prevalent in loose ash relative to the pelleted form. Increased base cation losses were attributed to rapid solubilisation of salt compounds and carbonates in the ash, and accompanying anions resulted in

leaching. P dissolution was attributed to the rise in soil pH and as a result the precipitation of Al and Fe phosphates. Phosphates are more susceptible to leaching due to it being an anion. Carbon and nitrogen dynamics were affected by a combination of wood ash and an additional N source; results showed increased CO_2 fluxes for the duration of the experiment. Research by Saarsalmi et al. (2012), experimental conditions described in Section 2.8, showed that wood ash applications combined with N fertiliser decreased exchangeable acidity in the organic layer up to 15 years after fertilisation. In addition, results showed increased CEC, base saturation and soil pH in the 5 cm - 10 cm and 0 cm - 5 cm mineral layer. A similar study by Saarsalmi et al. (2001), based on the effects of wood ash applications on soil properties in Scots pine and Norway spruce stands, found that wood ash applications increased the cation exchange capacity of the soil on all sites. Ash applications of 3 t ha^{-1} were made on the experimental area and soil and mineral samples were taken at 7 and 16 years after application for testing. CEC and base saturation increases were observed along with decreases in exchangeable Al in soil layers. The application of combined pulp/paper residuals and wood ash on *Eucalyptus grandis* plantations can result in Ca:K soil imbalances after two years, and as a result induce acute foliar K deficiency (Guerrini et al., 2000). The authors recommended a possible balancing K source to offset this potential imbalance.

Wood ash additions on agricultural soils can rapidly increase soil electrical conductivity following application (Perucci et al., 2008). Perucci et al. (2008) investigated the changes in physico-chemical and biochemical properties of soil following wood ash additions on an agricultural soil. Wood ash additions of variable particle sizes and two application rates of 5 and 20 t ha^{-1} were tested. Electrical conductivity, microbial biomass and hydrolytic activity were monitored up to 24 months following application and results indicated a significant relationship for the type of wood ash and sampling time. This interaction initially increased electrical conductivity and soil pH substantially following wood ash application; more so at high ash doses. Soil properties reverted back to the initial state after 12 months and were evident of a short term effects of wood ash on particular soils. Microbial responses for this study are explained in Section 2.8.

2.4.3 Growth response

The growth responses succeeding wood ash applications are not well documented, but positive and negative responses have been noted (Demeyer et al., 2001). Saarsalmi et al. (2012) found that wood ash applications co-applied with urea-N fertiliser can increase pine forest basal area increment up to 30 years after application. Scots pine trials were established in Northern Finland and fertilised with three wood ash application rates of 1, 2.5 and 5 t ha^{-1}. The application of wood ash increased the growth response to urea-N, but the response essentially stopped at 30 years of age. The application of a combination of wood ash and paper sludge on *Eucalyptus grandis* stands in Brazil showed increment increases in the range of 38 % - 64 % in volume up to 5 years after application. Increases of 75 % more volume were found in an oxisol and increments were compared to the response of chemical fertiliser. Soils used in the experiment were an oxisol, sandy oxisol and entisol; all three soils contained a substantial sand content (Guerrini et al., 2000). Similar trials in Brazil documented a positive growth response 6.5 years following wood ash application on *Eucalyptus grandis* stands. Ash application rates exceeding 15 t ha^{-1} did not yield additional increased growth (Gonçalves and Moro, 1995). The positive growth response from the wood ash applications was attributed to the increased soil availability of P, K and B following application (Demeyer et al., 2001). Growth and biomass responses from wood ash in nutrient poor sandy soils can result in reduced height growth in *Pinus abies* (Norway spruce) at application rates exceeding 5 t ha^{-1}, but greater root biomass and stem growth can be achieved at low ash application rates in the range of 2.5 - 5 t ha^{-1} (Mandre et al., 2004). The application of wood ash on N deficient and less fertile sites may result in reduced growth responses relative to wood ash treatments with a supplementary N source on more fertile sites (Jacobson, 2003).

2.5 Trace elements and base cations

Wood ash used as a soil ameliorant can provide the soil with micronutrients like Zn and B (Patterson, 2001). Increases in soil pH from wood ash additions can increase soil exchangeable Ca, Mn, Mg, K and decrease exchangeable Al and Fe (Naylor and Schmidt, 1989). The application of 11 t ha^{-1} wood ash on a N limited sandy soil can lead to increased leaching of base cations shortly after application in loose and

pelleted form, leaching is more prevalent in loose ash (Gómez-Rey et al., 2013). The authors studied the effects of wood ash application on nutrient leaching, soil C and N dynamics and fertility in a *Pinus pinaster* stand. Loose wood ash applications increased the leaching of Ca, Mg, Na and K in the 0-10 cm soil layer and resulted in increased nutrient availability and reduced soil acidity to a depth of 10 cm from the soil surface. Wood ash incubation studies by Ohno and Erich (1994) found a ten-fold increase in divalent Ca and Mg cation concentrations after 4 weeks. After the anticipated 72 week period, Ca and Mg concentrations were 15 and 22 times higher than the control. The substantial increases were attributed to the steep increase in soil pH due to the neutralising effect caused by wood ash additions and can be explained by the release of Ca, Mg, K and Na from the ash.

2.6 Soil depth and downward transmission

Gómez-Rey et al. (2013) observed leaching of base cations and soil P along with changes in soil C and N dynamics from wood ash applications at a depth of 10 cm from the soil surface. Experimental conditions are described in Section 2.5. Saarsalmi et al. (2001) studied the effects of wood ash on soil chemical properties of forest soils. The study was done on two contrasting wet and dry sites, planted with *Pinus sylvestris L*. and *Picea abies* (Norway spruce). Results indicated a pH rise of 0.6 - 1.0 units in the humus layer 16 years after application and a 0.2 - 0.3 pH unit increase in the mineral soil for the same duration. No significant increases in soil pH were recorded seven years after application for the mineral soil. The findings of Saarsalmi et al. (2001) provide further understanding with regard to the slow downward transmission of wood ash in certain soils; the reaction of wood ash is more likely to take place in the humus and topsoil layer. Williams et al. (1996) reported minor changes in elemental concentrations at a soil depth of 45 cm following wood ash applications and concentrations returned to initial levels (control treatments) within a period of 60 weeks. An ability to predict the downward movement of ash and its potential reaction with soil up to a certain depth is important as it will determine the quantity of soil reacting with any applied ash.

2.7 Charcoal content

Combustion temperatures for charcoal production range from 300 °C – 500 °C. Charring of *Eucalyptus camaldulensis* at 450 °C can result in a charcoal recovery by weight of 32 % and charcoal C content of 46.3 %. Charring *Eucalyptus grandis* at 470 °C can result in a charcoal recovery rate of 33.8 % and a charcoal carbon content of 80.7 % (Glaser et al., 2002). Pitman (2006) suggests biomass is burnt between 500 °C – 900 °C for maximum nutrient preservation and minimum heavy metal content in wood ash. Industrial boilers typically burn above 1000 °C (Etiegni and Campbell, 1991).

The combustion of biomass material produces mixed wood ash and can contain traces of charcoal. The presence of charcoal in wood ash can facilitate the slow release of nutrients and aid in circumventing the risk of depleting forest soil nutrients, and it can potentially act as a long term source of nutrients and organic matter (Santalla et al., 2011). Santalla et al. (2011) studied the effects of mixed wood ash application on *Pinus radiata,* applied independently and with an additional P source four years after application. It was found that the presence of charcoal could reduce P availability. Charcoal can absorb organic and inorganic P in its pores and lead to the formation of sparingly soluble phosphates; phosphates are subsequently released into the soil (Laird et al., 2010). Charcoal contains a high C: N ratio and could potentially reduce N mineralization and subsequently N uptake in plants (Steiner et al., 2008). This could be attributed to the fact that charcoal contains highly resistant C molecules, these molecules provide a poorly decomposable C substrate. This results in decreased C mineralization of soil carbon and subsequently reduced carbon-cycling in the soil (Liang et al., 2010). Residuals containing a high carbon content could additionally result in N immobilisation by soil microbial activity; micro-organisms utilize nitrogen to offset their internal C: N ratio (Goodwin & Burrow, 2006 and Matysik et al., 2001).

Wood ash consists of variable concentrations of P, K, Ca, Mg, Na and smaller amounts of N and organic matter. Organic matter content varies according to the degree of combustion. Higher combustion temperatures improve exchange properties and the surface area of charcoal (Glaser et al., 2002). Guerrini et al. (2000) suspected the presence of charcoal could explain the high organic matter

content in wood ash. The organic matter increase from charcoal does not necessarily contribute to the nutrient content of wood ash. An experiment based on the effects of wood ash on plant and soil nutrient characteristics established nutrient concentrations in charcoal can be low and at times insignificant (Park et al., 2004). Park et al. (2004) made annual wood ash applications for 3 years on coppiced willow (*Salix purpurea*), application rates of 10 and 20 Mg ha^{-1} were tested. Results found that the variability of charcoal in wood ash can range significantly and for the 3 year experimental period (1992, 1993 and 1994) the charcoal proportion of wood ash was approximately 26%, 16% and 34% respectively.

The effect of charcoal on nutrient dynamics and soil microbiology is strongly affected by the charcoal quantity applied. In a review by Glaser et al. (2002) on the amelioration of physical and chemical properties of highly weathered soils in the tropics with charcoal; it was noted that charcoal increases nutrient retention and crop production. The authors additionally investigated the effect of charcoal on soil microbiology and vice versa. The application of increasing amounts of charcoal can have similar results on soil microbiology as fly-ash application and effectively reduce microbial respiration, populations and enzyme activity. The increased nutrient content, retention capacity and reduced leaching is a product of the slow biological oxidation of charcoal which leads to the formation of carboxylic groups that provide cation exchange sites. The oxidation of charcoal thus produces organo-mineral complexes that lead to increases in CEC. Research has shown that charcoal is mineralized slowly into the soil environment (Glaser et al., 2002).

2.8 Soil microbial biomass; ectomycorrhizael populations, soil C and N microbiomass

Microbial communities are highly responsive to soil pH changes (Saarsalmi et al., 2012). Wood ash applications combined with an additional N source can potentially increase soil C and N microbial biomass and rate of C mineralization and subsequently lead to increased CO_2 production (Saarsalmi et al., 2012). The authors studied the effects of wood ash and urea N fertiliser on soil chemical properties, soil microbial biomass and stand growth. Separate and combined application rates of 1, 2.5 and 5 Mg ha^{-1} wood ash and nitrogen were made to *Pinus sylvestris* stands and monitored for 30 growing seasons. The authors concluded that wood ash in

combination with an N source affects soil chemical properties and C and N cycling microbial processes for a considerable period, 30 years in this study. Similar results were found by work previously done by Saarsalmi et al. (2010). The effects of wood ash and agricultural lime on certain ectomycorrhizal fungi species are not too different at times (Erland and Söderström, 1990). Erland and Söderström (1990) studied the effects of liming on ectomycorrhizal fungi species on *Pinus sylverstris L*. seedlings. Results showed variable seedling growth within treatments, but only a single ectomycorrhizal species (*Paxillus involutus*) was affected by the raised soil pH and resulted in increased infection of *Pinus sylverstris L*. seedlings. No significant differences for wood ash and agricultural lime treatments were found.

Wood ash applications on acidic and N rich peat soils can increase cellulose decomposition rate and bacterial populations after application, and as a result increase soil respiration rate (Pitman, 2006). Wood ash applications can increase ectomycorrhizal mycelium populations 2.4 times following application (Hagerberg and Wallander, 2002). Hagerberg and Wallander (2002) studied the impact of intensive harvesting and wood ash applications on external ectomycorrhizal mycelium populations. Results showed that wood ash amendments can increase ectomycorrhizal fungal biomass by approximately 2.4 times in a 13 month period. No significant effects were observed for the control and intensively harvested plots. Increases were observed in autumn, thus implicating seasonality as a determining factor for the effects of wood ash on EM populations. A review by Pitman (2006) on use of wood ash in forestry revealed that increases in soil respiration without changes to soil fungal biomass have been observed. Soil respiration increases were attributed to increased soil pH and mineralization of organic matter. A literature overview conducted by Demeyer et al. (2001) found that different wood ash application rates do not necessarily affect the microbial community as relative to fire treatments; fire significantly reduced microbial biomass. Wood ash and fire treatments were done on Scots pine trials and in addition found that fungi is more severely affected by high wood ash application rates than bacteria. Aronsson and Ekelund (2004) recommends the use of a more stabilised form of wood ash, such as a pelleted form to avoid potential damage on organisms and shock effects induced by high pH changes.

The effects of wood ash amendments on forest flora growth, fungi and soil fauna vary considerably. The variability revealed in studies is due to abiotic factors, site fertility, wood ash properties, different time scales of various studies (Aronsson and Ekelund, 2004), initial soil pH and soil buffer capacity.

2.9 Environmental challenges

This section provides an in-depth look at the environmental challenges and possible damaging effects of wood ash on fauna, flora and water sources.

2.9.1 Heavy metals

Wood ash contains variable concentrations of heavy metals and could potentially harm the environment, humans and several animal and plant species living in it. Heavy metals and trace elements like B, Zn, Cd, Se, Co, Hg, Pb, Ni, Cr and Cu have been reported, refer to Table 2.1 and 2.2 (Aronsson and Ekelund, 2004; Bird and Talberth, 2008; Demeyer et al., 2001; Elliot and Mahmood, 2006; Patterson, 2001; Pitman, 2006; Saarsalmi et al., 2012). The presence of heavy metals and other possible contaminants are acknowledged by all authors, but discrepancies are prevalent regarding their concentrations and reactivity. Heavy metal content increases from bottom to fly ash and bottom ash is better suited for land application (Demeyer et al., 2001 and Elliot and Mahmood, 2006).

High wood ash application rates may be detrimental to the environment due to the presence of cadmium (Patterson, 2001). Patterson (2001) additionally stated if ingested, cadmium can induce liver and kidney damage due to bioaccumulation of cadmium in plants and animal tissue. Relative to coal ash, wood ash may contain lesser quantities of potentially hazardous components such as arsenic, cadmium and selenium and is better suited to be used as a soil ameliorant (Elliot and Mahmood, 2006 and Bird and Talberth, 2008). Literature reviewed by Patterson (2001) revealed the solubility of heavy metals and trace elements from wood ash like cadmium and zinc can increase as soil pH values decrease below 6.5 units. Additionally solubility for both elements can increase substantially once pH values are reduced to below values of 5.5 – 6.0 units. The presence and chemistry of cadmium is to a great degree determined by soil pH. It can be adsorbed by clay minerals, carbonates and hydrous oxides of iron or manganese. Furthermore it can be precipitated either as a

carbonate, hydroxide and phosphate (McLean and Bledsoe, 1992). McLean and Bledsoe (1992) specified that under acidic soil conditions Cd solubility increases and little adsorption takes place by colloids, hydrous oxides and organic matter. At soil pH values greater than 6, cadmium is adsorbed in the soil solid phase or is precipitated (concentrations in solution are thus reduced). It is essential that the reaction of cadmium to diverse soil conditions is understood, as many South African forest soils have pH values lower than 5. Similar findings were made by László et al. (2009); the solubility of heavy metals in wood ash is low and as a result uptake by roots is hindered. The presence of cadmium in wood ash may decrease the colonization of ectomycorhizae and microbial activity (Aronsson and Ekelund, 2004). Research by Saarsalmi et al. (2012) on the effects of wood ash on soil properties, soil microbiology and Scots pine growth, experimental conditions described Section 2.8, reported elevated concentrations of Ca, Mg and K in the wood ash–nitrogen trials 30 years after application. Results did not show increased Cd concentrations in the organic layer, and Cd and Pb concentrations were not affected by the application of wood ash. Similar trials in Finland reported increased Cd levels, but the increases were minute and ranged within naturally occurring Cd levels. A review by Pitman (2006) states the adsorption of Cd into soils is governed by soil pH, organic matter and hydrous oxide content. Wood ash with high Cd concentrations has a significantly reduced leaching potential due the capacity of wood ash to bond with hydrous Mn oxides and hydrous iron oxides; wood ash contains significant concentrations of Mn.

Aronsson and Ekelund (2004) recommends extreme caution be followed if wood ash additions are made to soils; the mobility of Cd, Al and Cs could result in environmental harm and it is crucial wood ash is analysed prior to application. In conclusion they recommend wood ash applications be site-specific as an alternative to general guidelines for wood ash application to forests.

2.9.2 Water contamination

Bio-toxic effects of wood ash can be divided into two categories; primary and secondary effects. Potential primary effects are derived from the chemical composition of wood ash, especially cadmium and secondary effects from the alkalinity of wood ash and the leaching of ions in soil, groundwater or other water bodies (Aronsson and Ekelund, 2004). Land application of pulp and paper residuals

can be done short of compromising groundwater quality; residuals have to be mixed properly and applied at acceptable rates. Previous studies on pulp and paper mill residuals and sludge applications showed that Na and NO_3–N leaching may pose a threat to groundwater (Catricala et al., 1995). A potential risk of P surface run-off remains after wood ash additions and could lead to contamination of water sources (Patterson, 2001). A three year study based on the effect wood ash application on water quality in a drainage basin indicated small increases in K^+, SO_4^{2-}, Cl^- concentrations and phytoplankton biomass. Similar tank experiments based on the immediate effects of wood ash on water quality exhibited increases in water pH, alkalinity, conductivity, P and Ca concentrations and a decrease in phytoplankton biomass (Aronsson and Ekelund, 2004).

The application of wood ash may have a slow downward transmission into soil horizons and has little effect on soil water (Williams et al., 1996). Williams et al. (1996) studied the effects of bottom ash from bark boilers on forest and water chemistry in the United States. Application rates of 11, 22 and 44 Mg ha^{-1} were made per rotation; groundwater samples showed minute changes and only significant changes were noted for Ca, K and SO_4. Groundwater remained unaffected for all application rates and was within the standards set for consumption.

2.10 Policy and legislation

The Fertilizer, Farm Feeds, Agricultural Remedies and Stock Remedies Act, 1947 (Act no. 36 of 1947) disregards the use of pulp and paper residues with high organic or nitrogen content as fertilizing agents. Fly ash is categorised as air pollution under the National Environmental Management: Air Quality Act (Act no 39. of 2004) (Department of Environmental Affairs, 2013). This would suggest the release of fly ash particulates into the atmosphere from smoke or wind. Legislation states the Minister of Agriculture has the authority to register any new agricultural substances for agricultural development if the proposed substance adheres to the standards stated in the Act (Department of Agriculture, 2008). Current legislation does not allow the use of pulp and paper residues as a fertilising agent or soil ameliorant due to reasons stated previously, however the use of wood ash (exclusively bottom ash) is not specified. It is essential to gain a comprehensive understanding regarding the classification of organic wastes, hazardous materials, compost and fertilizer

according to South African Legislation. In the National Organic Waste Composting Strategy of 2013, the National Environmental Management: Waste Act (Act No. 59 of 2008) organic waste is defined as waste generated from garden, food and wood sources. The same act defines hazardous waste as waste containing organic and inorganic elements or compounds that can as result of its physical, chemical or toxicological properties have a detrimental effect on health and the environment. The Fertilizers, Farm Feeds, Agricultural Remedies and Stock Remedies Act (Act No. 36 of 1947): GNR 732 Regulations Regarding Fertilizers defines compost as a stabilised substance of plant or animal origin that is fully decomposable, has no additional nutrients added or any substances/elements that has detrimental effects to man or the environment. The same act defines fertiliser as any substance proposed to be used for improving or maintaining plant growth or soil productivity (Department of Environmental Affairs, 2013). The National Environmental Management: Waste Act (Act No. 59 of 2008) defines a by-product as a material produced as part of a process intended to manufacture another substance or product and retains properties of the original source or virgin material (Department of Environmental Affairs, 2013). Wood ash is produced as a by-product of pulp and paper mills, but chemical composition is determined by a range of conditions as stated in Section 2.2.1. A review by Pitman (2006) on the potential use of wood ash in forestry states Cd, As, Hg and Pb concentrations should not be problematical if bottom ash is used. With conservative wood ash application rates there is virtually no environmental risk, provided pure woody material is used (Demeyer et al., 2001) and the buffer capacity of the soil is not exceeded. The allocation of certain organic materials, particularly bottom ash, as a potential fertiliser source is ambiguous according to current South African law.

Tables 2.4 and 2.5 present the soil screening values and standard levels for heavy metals according to the South African legislation and other countries. Heavy metal and metalloid soil screening values according to the South African Department of Environmental Affairs' National Environmental Management: Waste Act, 2008 (Act no. 59 of 2008) are presented in Table 2.4. The standards outlined in Table 2.4 provide the minimum concentrations for assessing environmental protection and subsequent actions for rehabilitation. Screening values are not absolute minimum values, but for screening purposes.

Table 2.5: South African soil screening values for metals according to the National Environmental Management: Waste Act, 2008 (Act no. 59 of 2008). (Department of Environmental Affairs, 2013).

Parameter	All land uses. Protective of the Water Resource	Informal Residential	Standard Residential	Commercial / Industrial	Protection of Ecosystem Health
Units	mg/kg				
Arsenic	5.8	23	47	150	580
Cadmium	7.5	15	32	260	37
Chromium (III)	46 000	46 000	96 000	790 000	n/a
Chromium (VI)	6.5	6.5	13	40	260
Cobalt	300	300	630	5 000	22 000
Copper	16	1 100	2 300	19 000	16
Lead	20	110	230	1 900	100
Manganese	740	740	1 500	12 000	36 000
Mercury	2	1	1	6.5	4.1

Nickel	91	620	1 200	10 000	1 400
Vanadium	150	150	320	2 600	-
Zinc	240	9 200	19 000	150 000	240

Herselman (2007) established that South African soils have high naturally occurring concentrations of Cr, Ni and Pb. Table 2.5 presents a summary by Herselman (2007) on the maximum permissible (tolerable) soil metal levels in different countries, expressed in mg kg^{-1}. Both the SA 1991 and the more recent SA 1997 heavy metal concentrations are guidelines regarding the allowable soil trace metal concentrations for South Africa. The SA 1991 guidelines are more realistic and comparable to other countries relative to SA 1997 guidelines. If the soil metal content exceeds the permissible amount, wood ash amendments should be avoided. The guidelines do not account for soil type and incorporate naturally occurring heavy metal concentrations.

Table 2.6: Maximum permissible soil metal levels for different countries in mg kg^{-1} (Herselman, 2007).

	EU	USA	Germany	Australia & New Zealand	SA 1991	SA 1997
As	-	21	-	20	2	2
Cd	1-3	20	3	3	2	2
Cr	-	1500	400	50	80	80
Cu	50-140	750	135	60	100	6.6
Hg	1-1.5	8.5	-	1	0.5	0.5
Ni	30-75	210	75	60	15	50
Pb	50-300	150	300	300	56	6.6
Zn	150-300	1400	300	200	185	46.5

The ambiguity of current South African legislation ascertains further insight and strategies are needed regarding this matter.

2.11 Conclusion: Literature Review

Wood ash is one of the main by-products of tree biomass combustion produced in pulp and paper mills and numerous boiler systems. It contains a range of macro and microelements in generous concentrations, but little or no nitrogen. Several studies have indicated wood ash and additional N sources greatly improve the growth of eucalypt and pine stands tested. Risk to the environment ensuing from the presence of heavy metals, dioxins and furans can be avoided by not using excessive application rates, and through an understanding of the soil chemical reaction with added ash materials, in particular heavy metals. It follows that a complete chemical analysis of wood ash needs to be done and current soil metal content and soil buffer capacity is known. On sensitive systems it is essential that pure wood ash; free of possible contaminants is used, because other sources of ash may lead to high dioxin, furan and heavy metal concentrations. The effects of wood ash on soil microbial activity varies; literature shows wood ash can largely increase microbial biomass on acid soils, but minimal effects or even decreases can be encountered (especially if soil pH is increased too drastically). The presence of heavy metals can lead to possible water contamination, risk is minimised with acceptable application rates and downward transmission of heavy metals is reduced significantly at higher pH values.

The disposal of wood ash on landfills in South Africa is costly and damaging to the environment. Several reviewed sources have shown wood ash can potentially be safely disposed of on plantation soils and more importantly replace nutrients removed from intensive tree harvesting systems. The liming effect induced from the presence of Ca, hydroxides, oxides and carbonates can potentially alleviate acidic soil conditions and improve pine and eucalypt growth, provided that soil pH is not raised too high. Tree growth and soil reactions to wood ash additions are likely to be site-specific. The diverse and opposing results obtained from international literature is indicative of the effects of factors such as initial soil pH, soil buffer capacity, climate, tree genotype, other soil characteristics and residue composition can have on the tree growth response to wood ash fertilisation.

3 Chapter 3: Material and Methods

3.1 Site description

3.1.1 Location

The trial site was established on the Zululand coastal plain approximately 7 km from Central Richards Bay (Kwazulu-Natal, South Africa) and positioned 12 km from the Indian Ocean. Site coordinates are 28°41'55.93" S and 32° 2'3.28" E and the site is located 64 m above sea level. The plantation is owned by Mondi and identified as the IONA plantation, compartment A014. Richards Bay has a sub-tropical climate with warm wet summers. Figure 3.1 shows the location of the study site In KwaZulu-Natal, South Africa. Soils in the region have low nutrient reserves and high nutrient turnover, thus allowing for highly productive plantation forestry. In addition, soils have a low organic C content (less than 0.5%), N content (less than 0.05%) and a clay percentage of less than 5 % in general (Smith and du Toit, 2005). Table 4.1, Chapter 4, presents soil chemical and physical properties, exchangeable acidity and pH values for soil samples collected at the trial site. During the reconnaissance survey, soil samples were bulked at 5 points within the trial site at a soil depth of 0 – 20 cm using an auger. When comparing data in Table 4.1, collected during the reconnaissance survey, with literature on the forest soils of the coastal plain, it is clear that the trial site is representative of the soils in the region (Smith and du Toit, 2005; du Toit and Oscroft, 2003).

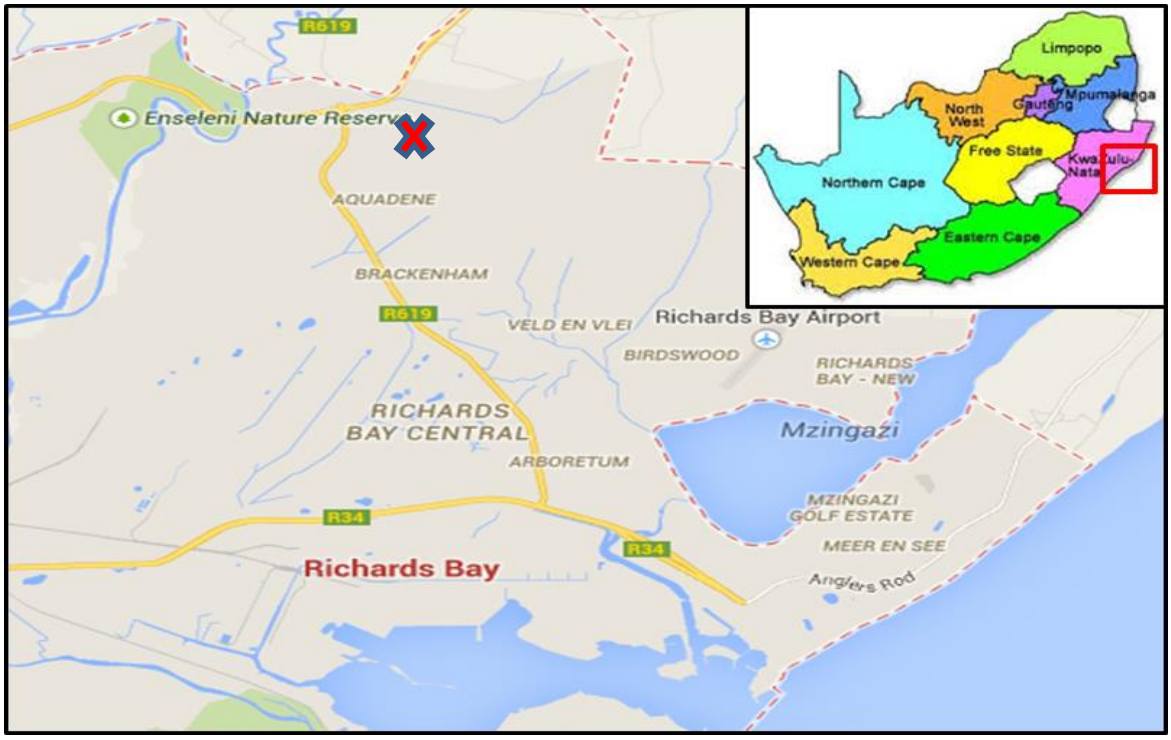

Figure 3.1: Map showing the location of the trial area in KwaZuku-Natal, South Africa. Adapted from Geology.com, 2014; Google maps, 2014.

3.1.2 Site history

The site was presumably occupied by Kwazulu-Natal coastal forest prior to the 1950's. It is most likely that the land would have been used for agricultural production such as sugarcane in the period from the 1950's to 1970's. It appears that the site had been afforested with Eucalypts since the 1980's. The last rotation of trees before trial establishment was a stand of Eucalyptus *grandis x urophylla* hybrid clones that was harvested at the end of 2012. It had been planted at an initial spacing of 2.4m x 3m and had a final planting density of 1389 trees per hectare.

3.1.3 Climate

Richards Bay Municipality and Airport weather stations recorded the climatic data used in this project and was provided by the South African Weather Service. Figures 3.3 and 3.3 graphically illustrate the mean minimum and maximum monthly temperatures and rainfall for Richards Bay for the last 25 years. Weather stations are located approximately 7.5 km and 6 km from the trial site. According to data from the last 25 years Richards Bay has a mean annual temperature and rainfall of 22 °C and 1221 mm respectively. The months of December - March have the highest temperatures and the warmest mean monthly temperatures range from 28.7 °C -

29.5 °C. July - August have the lowest mean monthly temperatures in the range of 13.2 °C - 16.8 °C.

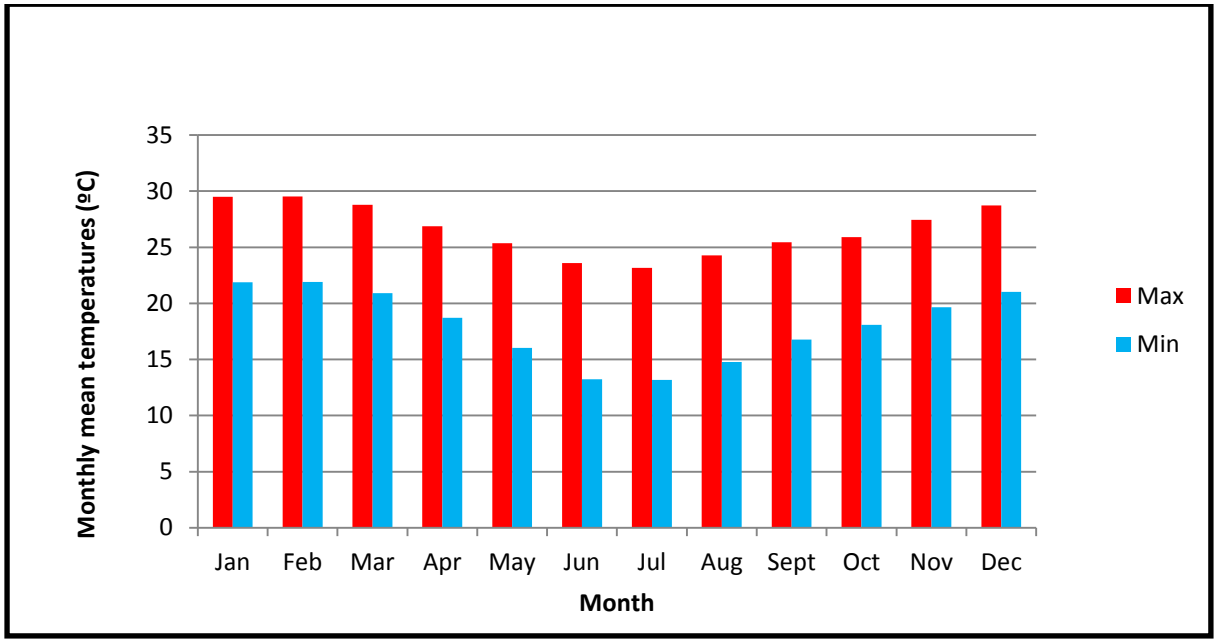

Figure 3.2: Mean monthly minimum and maximum temperatures for Richards Bay.

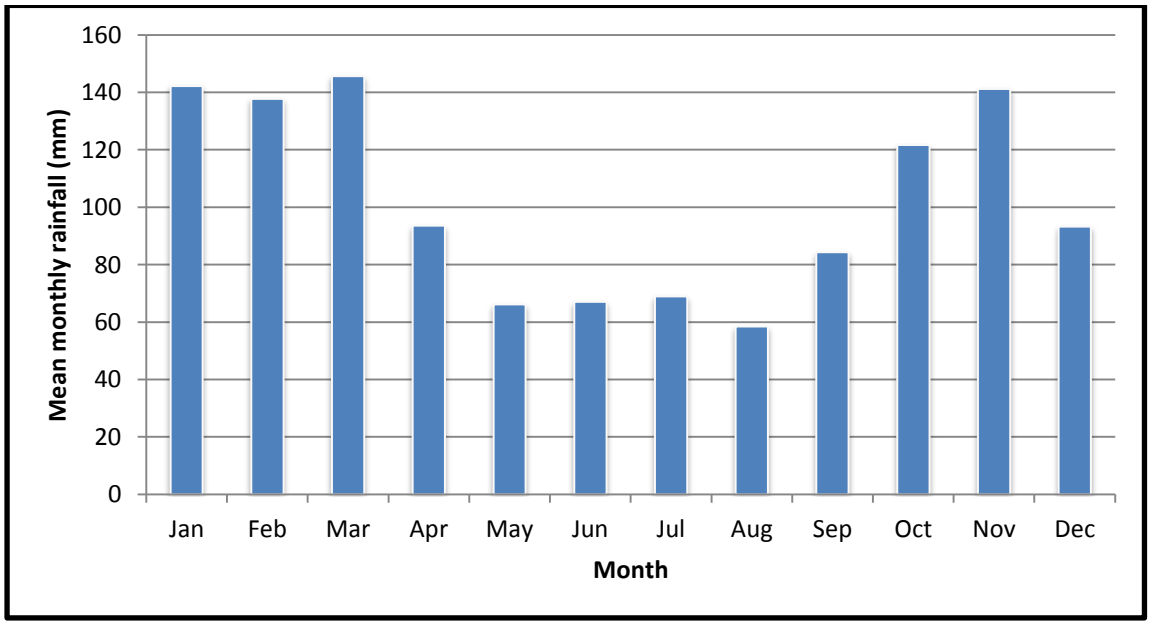

Figure 3.3: Mean monthly rainfall for Richards Bay.

3.2 Field experimental design

The experimental methodology is divided into two sections; laboratory (Section 3.3) and field work. A trial area of 2.5 hectares was selected for the field study. The study was designed to test the interaction of two key variables; wood ash application rate

and fertiliser variety. Four wood ash application rates and three fertiliser varieties were tested in a 4x3 factorial design. The allocations of treatments to plots were randomised using the Microsoft (MS) Excel 2010 RAND function. The experiment was replicated 6 times and the allocations of replications were also randomised within the blocks to produce a total of 72 plots. Treatment combinations and replications were as follows:

$A_{0,1,\ 2,\ 3}$ - Ash application rates: Wood ash application simulated per plot; 0, 300, 600 and 1200 kg.ha^{-1} scaled down to values of 0, 9, 16 and 31 kg wood ash per plot.

F_0 - No fertiliser application

F_{CRF} - Controlled release fertiliser (CRF): Application rates of 320 g per seedling CRF were applied to the designated randomised plots. CRF contained 80 g N and 40 g P. Section 3.4.4, Table 3.1, presents application rates of individual nutrient elements in the CRF prescription blend.

F_{CV} - Conventional fertiliser (CV): Application rates of 150 g per seedling Ammonium Sulphate Nitrate fertiliser was applied to the designated randomised plots. The CV prescription blend contained 40 g N.

Plots were planted at the standard spacing of 2.4m x 3m. Each plot comprised of 7 x 7 rows of trees, the outer row represented a buffer row. At sampling and data collection only the inner 5 x 5 rows were sampled and analysed for the trial. Each inner plot had an effective area of 180 m^2.

3.3 Soil buffer capacity

In order to determine the lime requirement and more importantly the wood ash application rates tested on the trial site, the soil buffer capacity had to be calculated. The soil buffer capacity was essential as it would primarily determine the amount of wood ash the soil could accommodate and the resultant soil chemical changes.

3.3.1 Soil sampling

During the reconnaissance survey five topsoil samples, representative of the trial as a whole were collected on the 21st August 2013, approximately 7.5 weeks (54 days) before trial establishment. Soil samples were randomly sampled at 5 points within

the trial area at a soil depth of 20 cm using a manual Dutch auger. Each sample was made up of a single auger bucket and was individually sealed in a plastic bag.

3.3.2 Soil analysis procedure

A comprehensive nutrient analysis was done on each soil sample to identify any properties or possible constraints that might affect the soil's ability to accommodate wood ash applications. The soil analyses results presented in Table 4.1, used to describe the site soil characteristics, were analysed at the KwaZulu-Natal Soil and Fertility Analytical Services, Department of Agriculture, Cedara. The soil chemical analysis procedure outlined in Section 3.5.2 was used for samples collected during the data collection period and all results are based on this methodology for consistency.

The following section describes the methodology used by Cedara Agricultural College Manson and Roberts (2000) for the five soil samples used to describe the soil properties of the site.

The soil samples were initially air dried at room temperature by evenly spreading the samples and forcing air over them.

Soil analysis was conducted on a volume basis and sample density was determined by measuring the mass of a 10 ml scoop for each soil sample and determining the sample density. Soil pH was measured by taking 10 ml of each sample and adding 25 ml 1M KCl solution to each sample. The suspension was then left for 30 minutes and after which a gel-filled combination glass electrode was used to measure the pH of each sample.

Extractable (1M KCl) Ca, Mg and acidity was determined by measuring 2.5 ml soil from each sample and adding 25 ml KCl solution to each sample. The suspension was subsequently stirred at 400 revolutions per minute for 10 minutes using a multiple stirrer. After 10 minutes the solutions were filtered using Whatman No. 2 paper and 5 ml filtrate from each sample was diluted with 0.0356 M $SrCl_2$. Ca and Mg contents were determined by atomic absorption. Extractable acidity was determined by measuring 10 ml filtrate from each sample and diluting them with 10 ml de-ionised water intermixed with 2-4 drops of phenolphthalein. The final product was then titrated with 0.005 M NaOH to determine the acidity. Extractable (Ambic-2)

P, K, Zn, Cu and Mn was determined by measuring 2.5 ml soil from each sample and adding 25 ml ammonium bicarbonate solution to each. The solution was subsequently stirred at 400 revolutions per minute for ten minutes using a multiple stirrer. Solutions were then filtered using Whatman no. 1 paper. Phosphorous content was determined using the molybdenum blue procedure described by Hunter (1974). Potassium content was determined by adding 20 ml de-ionised water to each solution and applying atomic absorption on a 5 ml aliquot of each filtrate. The remaining filtrate was used to do atomic absorption to determine K, Zn, Cu and Mn content.

The Effective Cation Exchange Capacity was determined as the sum of KCl-extractable Ca, Mg, acidity and Ambic-2 extractable K. The percentage acid saturation was determined as the extractable acidity multiplied by 100 divided by the sum of Ca, Mg, K and extractable acidity. Organic carbon percentage and clay content was determined using near-infrared spectroscopy (Manson and Roberts, 2000). Near-infrared reflectance was used on the crushed air dried samples. Measured sample density was included for clay content estimation and conversion of volume based concentrations to mass a basis. Total carbon and nitrogen content for each sample was determined according to the Automated Dumas combustion method. Soil samples were weighed in crucibles and 0.5 g vanadium pentoxide was added as a combustion catalyst. The crucible was subsequently burnt in oxygen at 1325 °C. Gases produced by the combustion process passed through two infra-red cells; the carbon (CO_2) produced was measured and total nitrogen (N_2) in a thermal conductivity cell.

3.3.3 Wood ash analysis procedure

Initially wood ash produced by the Richards Bay pulp and paper mill boiler system would have been used in the study, but due to boiler system upgrades an alternative wood ash source was needed. It was proposed that wood ash derived from the combustion of tree biomass be used, free of chemical additions from the paper production process. DukuDuku sawmill, located 10 kilometres from St. Lucia (KZN), was used as an alternative wood ash source; wood ash was produced by burning Eucalyptus saligna waste generated during wood processing. It is important to note that wood ash produced by pulp and paper mill boiler systems might contain higher concentrations of potential contaminants and heavy metals relative to the wood ash

produced by a sawmill. The study serves as the first step in providing evidence for the potential use of clean wood ash (produced from biomass) on plantation soils in Richards Bay.

The sawmill burns eucalypt waste and small amounts of leftover bark produced during timber processing. Timber was untreated. Figure 3.4 presents an image of the waste materials produced by DukuDuku sawmill and Figure 3.5 a closer image of the ash used in the trial after combustion. Ash samples were collected periodically at three intervals to account for variation in the wood ash.

Figure 3.4: Biomass burnt by DukuDuku sawmill.

Figure 3.5: Biomass ash produced by DukuDuku sawmill.

Five wood ash samples were selected for full nutrient analysis and a Calcium Carbonate Equivalent test (CCE). Ash samples were sampled at different times and from different waste streams, this was done to account for variation. The mean CCE or Neutralising Value for the samples was determined as 73.5% by the Cedara Agricultural College using the A.O.A.C method, indicating that 100g of wood ash would neutralise the same amount of acidity than 73.5 g of pure $CaCO_3$.

Plant material is usually prepared for analysis by ashing and filtering. Although the analysis being conducted on wood ash, ashing of the material was required as carbon content was high for the acquired ash material (Table 4.1). Half a gram for each sample was weighed out and into a 100 ml beaker and subsequently oven-dried for two hours at 105 °C. After the drying period, each beaker was reweighed. Each sample was left to cool off and wetted with distilled water. Two ml of concentrated HCl was added to each sample. Each sample was then gradually dried on a water bath in a fume cupboard with an extractor fan. A 25 ml freshly prepared 1:9 HCl solution was subsequently added to each sample using a Fortuna Optifix and individually stirred with a rubber stirring rod. The rod was rinsed with distilled water following each stirring. Lastly each sample was filtered through Advantec 5B: 90 mm paper into a clean rack of sample cups. The filtrate for each sample was diluted with de-ionized water to a ratio of 5:20. The elemental concentrations for Ca,

Mg, K, Na, P, Cu, Zn, Mn, Fe and Al were determined using the ICP-OES (Inductively Coupled Plasma Optical Emission Spectroscopy).

Total C, N and S were determined on fresh wood ash samples using the Dumas dry combustion method. Samples were burnt at 1350 °C in a furnace, converting elemental carbon, nitrogen and sulphur into gasses. Subsequently the gasses were homogenised and passed through infrared detection cells (CO_2 and SO_2) and a thermal conductivity cell (N_2 and NO_x) to measure the elemental C and N contents.

Three wood ash samples, sample at different time intervals were used for heavy metal analysis. Wood ash heavy metal content was measured using methodology based on Clesceri et al. (1998). Three wood ash samples were reduced to 5 ml and digested on a sand bath using 20 ml nitric acid (55%) and 5 ml hydrogen peroxide; this was done until the volume was reduced to less than 10 ml. All three samples were subsequently made up to 10 ml with distilled water and prepared for analysis. Samples were analysed for Cadmium, Mercury, Arsenic, Lead and Chromium using an ICP-AES (Inductively Coupled Plasma Atomic Emission Spectroscopy). Samples were transferred to a 20 ml ICP (Inductively Coupled Tube) and analysed using the ICP-AES. The wavelengths recorded for Cadmium (214.4 nm), Hg (184.9 nm), As (193.7 nm), Pb (220.4 nm) and Cr (267.7 nm) served as an approach to measuring the heavy metal content for each sample.

3.3.4 Soil buffer capacity

Following the soil analysis, soil pH irregularities were found in soil sample 2 and it was decided to exclude the sample from the pilot study. The remaining 4 soil samples were dried for five to six hours at 60 °C. Subsequently, for each soil sample 8 sub-samples of 100 g each were made up in perspex containers, thus amounting to 32 sub-samples. The buffer capacity of a soil refers to the ability of a soil to resist a change in pH after an alkaline or acidic material is applied (Kissel et al., 2011). The buffer capacity was determined according to an adaptation of the methodology used by Aitken et al. (1990); the methodology entails the mixing a soil with a buffered solution and recording the change in pH. Aitken et al. (1990) calculated the soil buffer capacity by fitting linear regression graphs for the relationship between recorded soil pH (x-axis) and rate of added $CaCO_3$ (y-axis). The author defines

buffer capacity as the change in quantity with intensity and therefore the reciprocal of the slope given by the linear regression is recorded as the buffer capacity of the soil.

Soil samples 1, 3, 4 and 5 were each subdivided into 8 sub-samples, each weighing 100 grams. Two sets of sub-samples (batch #4 and #5) were treated with $CaCO_3$ applications in the following range: 0, 30, 60, 90, 100, 200, 300, 400 mg. The $CaCO_3$ used in the pilot study was in powder form. The sub-samples sets from batch #1 and #3 were treated with wood ash. Wood ash quantities were adapted to amount to the same CCE value as the pure calcitic lime. The 0, 30, 60, 90,100, 200, 300, 400 mg $CaCO_3$ and adapted wood ash quantities were individually dissolved in 1 litre flasks of distilled water using a magnetic stirrer. Liming agents ($CaCO_3$ and wood ash) were added as completely dissolved solutions, due to wood ash and $CaCO_3$ not being very soluble in water. 25 ml of each lime and wood ash solution was added to the 100 g soil samples and incubated at room temperature for 3 days. Samples were dried for 5-6 hours at 60 °C following the incubation period. Soil samples were reduced to 20 g and 50 ml 1M KCl solution was added to each sample. Samples were placed in the automatic shaker for 15 minutes and subsequently pH measurements were taken using the Metrohm 744 pH-meter. The unit conversion from milligrams buffered solution per litre to added milligrams per kilogram soil for the liming materials was mathematically adjusted from 0, 30, 60, 90, 100, 200, 300 and 400 mg/l to 0, 6, 12, 18, 20, 40, 60 and 80 mg/kg. Four linear regressions were done for each soil in the pilot study using Microsoft (MS) Excel 2010.

3.3.5 Lime requirement

The lime requirement was determined using an adaptation of methodology by Kissel and Vendrell (2012). The lime requirement was used (to adjust soil pH to a predetermined value) as an index of how much wood ash can be applied to a soil before it exceeds the predetermined value. Equation 1 was used to determine the lime requirement for IONA AO14. It incorporates the lime buffer capacity, target and initial pH, lime purity and soil depth. A target pH of 6.5 was selected for this experiment. The average lime requirement for the four soil samples was used as the lime requirement for IONA A014. The lime requirement was determined as 250 kg/ha. As expected, the lime requirement was initially very low and it was noted the site had a high pH at initial soil analysis, refer to Table 4.1 for initial pH values.

Equation 1: LR (mg $CaCO_3$ kg^{-1}) = LBC × (Target pH –Initial pH) ×3×1.5 (Kissel & Vendrell, 2012).

Where:

1. LBC has units of ppm, and a factor of "2" is used in the original equation to convert to lbs/acre. Converting lbs/acre to kg/ha uses a conversion factor of 1.14. A factor of 3 was used in this project: The soil depth factor, outlined in point 2, was incorporated into the conversion factor and multiplied with 2.28 (1.14*2) and amounted to 3.03.
2. Soil sampling was done at a depth of 20 cm. The conversion from ppm to lbs/acre is for a 6 inch (15.24 cm) soil depth. Thus for a conversion from a 6 inch to 8 inch (20.32 cm) soil a factor of 8/6 (20.32/15.24) is used. A soil depth factor of 1.33 is used in this project.
3. The LBC incorporates pure $CaCO_3$; "1.5" is used for agricultural lime with a lower purity. The factor of 1.5 retained in the equation due to uncertainty regarding the depth wood ash could react with the soil. The calculated ash requirement value was used as the basis for the lowest ash application rate (Section 3.3.6).

3.3.6 Ash requirement

The ash requirement was determined using methodology by Lickacz (2002) from Alberta Agriculture Food and Development. Equation 2 incorporates the CCE of the wood ash, percentage moisture and lime requirement to determine the wood ash requirement for IONA A014. The average moisture percentage for all ash samples, presented in Table 4.2, was incorporated into the equation. The ash requirement was determined as 341 kg/ha. This value was then used to determine the application rates tested in the experiment. Wood ash treatments used in the trial were based on the base rate, as well as two and four times the amount to test the maximum amount of ash that could be applied before growth suppression and possible soil contamination is induced. Applications rates of 0, 300, 600 and 1 200 kg/ha were used at trial establishment.

Equation 2: Ash Requirement = $\frac{100}{CCE\ of\ ash} \times \frac{100}{100-\%H2O} \times \frac{Lime\ requirment(\ kg/ha)}{1}$ (Lickacz, 2002)

Where:

1. CCE of ash refers to the Calcium Carbonate Equivalent of the wood ash
2. $\%H_2O$ refers to the percentage moisture in the ash.
3. Lime requirement as provided by the soil testing laboratory (Determined in Section 3.3.5).

3.4 Trial establishment

3.4.1 Site requirements

It was initially decided that the site intended for ash applications had to adhere to certain requirements to minimize potential environmental risk and wood ash loss. The wood ash had a powdery texture and could potentially be transported offsite by strong winds and be deposited on natural vegetation or riverine areas. Residues on IONA A014 were burnt following harvesting; a common practice by South African forest companies. The site had an unusually high slash load following burning and was attributed to slashed coppice material from old stumps and pre-burning in unfavourable conditions, thus resulting in low intensity fires and a partially slash retained site. Coppice shoots were measured to be approximately 2m in height before it was slashed and distributed fairly uniformly across the site as shown in Figures 3.6 and 3.7.

Figure 3.6: Height of slash on experimental site (Pictured: Denis Oscroft).

Figure 3.7: IONA A014 trial site–showing slash loads at time of establishment.

3.4.2 Wood ash application

Wood ash applications were done on the 22nd October 2013, 8 days after planting. Due to low initial wood ash application rates the risk of seedling scorch from the wood ash was low. The plots were divided into sub-plots of 2.4m x 3 m (i.e. the area between 4 planting pits). Ash applications were measured out per sub-plot to ensure the application was fairly uniform across the entire plot. Cup sizes for ash application were adjusted to 250, 444, 861 grams. Cup sizes, representative of the ash application rates, were selectively changed during ash application for each plot. Personnel were instructed to broadcast ash between individual trees within plot boundaries; care was taken to avoid ash application on seedlings and overstepping into adjacent plots.

3.4.3 Planting, blanking and weeding

The site was planted on 14th October 2013 by Mondi contractors. The site was blanked on the 23rd October 2013, a day following treatment application to ensure 100% stocking. First ring weeding was done on the 28th January 2014 by Mondi contractors. Early mortality count was done on the 17th February 2014. Coppice reduction was done on the 24th and 25th February 2014.

3.4.4 Fertiliser application

Two fertilisers were chosen for this experiment; a CRF (controlled release fertiliser) and CV (conventional fertiliser). As stated in Chapter 2, the effect of wood ash on

soil chemistry and nutrient availability is site specific and can affect the soil immediately or gradually following application. Wood ash contains little or no nitrogen and an additional source of nitrogen is needed to ensure balanced nutrition. Thus the use of both fertilisers in combination with varying wood ash rates could shed light on the interaction between the type of fertiliser (immediate or gradual nutrient availability) and the amount of ash applied. Controlled release fertiliser was produced and mixed by Haifa Chemicals Ltd. The controlled release fertiliser had a polymer coating that released 25% of nutrients by 2 months after application and 75% at 8 months (Table 3.1). The fertiliser comprised of 80 and 20 grams nitrogen and phosphorous and 320 g CRF was applied per seedling. The CV fertiliser contained 40 g N as ASN.

Table 3.1: Application rates of nutrient elements using the CRF prescription blend at 320 grams per seedling.

	N	P	K	Ca	Mg	S	B	Cu	Fe	Mn	Mo	Zn
Units	g/tree					mg/tree						
Elemental application												
CRF	80	20	0	10	1	12	0	90	128	96	0	1152
CV	40											

Cups were also modified for fertiliser application to 320 g CRF and 150 g CV. CRF was placed approximately 10 cm from the seedling in an adjacent furrow created with spades (Hans, 2013). The CV fertiliser was placed one spade length (approx. 30 cm) away from the seedling in a furrow made perpendicular to the seedling. CV application was done at a greater distance from the seedling due to increased risk of scorch (du Toit and Oscroft, 2013), as illustrated in Figure 3.8. Both the CRF and CV fertiliser application rates were based on the optimum levels obtained for that specific fertiliser type under field trial conditions (du Toit and Oscroft, 2003; Hans, 2013).

Figure 3.8: CV fertilizers application.

3.5 Data collection and analysis

3.5.1 Soil sampling

Soil samples were collected at 4 and 8 months after establishment; first soil sampling was done on the 18th February 2014 and the final sampling on the 10th June 2014. Soil samples were collected from the soil surface to a depth of 10 cm using a Beater Auger. The Beater Auger allowed for multiple sampling per plot; 10 samples were collected per plot and stored in plastic bags. Figure 3.9 shows the beater auger used for topsoil sampling. The project intended to investigate what quantity of wood ash could be applied to the soil without any detrimental effects, thus only plots treated with the fixed wood ash application rates were selected for soil sampling. Fertiliser treatments were excluded.

Figure 3.3.9: Beater Auger used for soil sampling during data collection.

3.5.2 Soil nutrient analysis technique

Soil samples were air dried and subsequently sieved through a 2mm sieve; this additionally allowed for the determination of the stone fraction for each sample. The pH for each sample was determined using 1M KCl. Chemical analysis procedures used: P content was determined using Bray II and extractable cations K, Ca, Mg and Na were extracted using 0.2 M ammonium acetate with a pH of 7. Extracted solutions were analysed for chemical composition and elemental concentrations by ICP-OES. Total P was extracted at 80 °C for 30 minutes using a 1:1 mixture of 1 M nitric and hydrochloric acid. P concentration was determined using the Varian ICP-OES. Extractable acidity for each sample was extracted using 1 M KCl and determined through titration with 0.05 M NaOH. Total C and N content was determined through high temperature combustion using the Leco Truspec® C and N analyser. Total soil NH_4^+ and NO_3^- concentrations were determined by extracting

ammonia and nitrate with 1 M KCl solution and subsequently measuring the concentration colorimetrically using a SEAL AutoAnalyzer 3. CEC was determined according to the methodology described by The Non-affiliated Soil Analyses Work Committee (1990); using 0.2 M ammonium acetate with a pH of 7. Samples were leached with 0.2 M K_2SO_4. Ammonia was extracted using 1 N KCl and afterwards concentrations were colorimetrically determined using the SEAL AutoAnalyzer 3 with a 15 mm flow cell and 520 nm filters. Field soil moisture was determined on a mass/mass basis; soil samples were dried overnight at 105 °C. The Bulk Density was determined by weighing 60 cm^3 for each sieved sample at 20 °C and expressing it as kg/m^3. Soil texture was determined by disaggregating the samples using sodium-hexametaphosphate (calgon) and ultrasound dispersion; the three sand fractions were determined by the methodology described by The Non-affiliated Soil Analysis Work Committee (1990). Silt and clay fractions were determined using an ASTM E100 (152H-TP) hydrometer at 20 °C.

Readily oxidisable oganic matter content was determined using the Walkley-Black method. Potassium dichromate and concentrated sulphuric acid were added to 0.5 to 1.0 g soil samples. Each solution was swirled and left to cool prior to adding water to stop the exothermic reaction. The amount of organic carbon was then spectrophotometrically measured (Schumacher, 2002).

3.5.3 Soil heavy metal analysis technique

Soil samples were sieved through a 2 mm sieve and subsequently dried. Extraction was done by measuring 5g of the sieved and dried samples and adding 20ml HNO_3 (55%) and 5ml hydrogen peroxide (30%); subsequently samples were placed on a heated sandbed for 8 hours and then filtered using Whatman no. 2 filter paper. Extraction was done using atomic emission with a Varian ICP-OES optical emission spectrometer. Heavy metal was measured according to each element's wavelength. Wavelengths for the selected elements are given at the end of Section 3.3.3.

3.5.4 Foliar sampling

Foliar sampling was done at 4 and 8 months after establishment; first sampling was done on the 19th February 2013 and second sampling on the 11th June 2013. Foliage samples were collected from each tree in the trial. Each plot was made up of 25 designated trees (excluding boundary trees) and the experiment as a whole

contained 72 plots. Four fully expanded leaves were sampled from each of the 25 trees per plot and sealed in plastic bags; subsequently bags were stored in a mobile cooling unit to prevent deterioration of samples.

3.5.5 Foliar nutrition and heavy metal analysis technique

Analysis was done to determine nutrient concentration following wood ash treatments to soils. Foliage samples collected were stored in a fridge at approximately 3 °C for four days after collection. Samples were analysed for nutritional status and heavy metal content subsequently. Samples were washed with a Teepol solution, rinsed with de-ionised water and then oven dried at 70 °C. Subsequently the dried samples were milled and ashed at 470 °C. A 50:50 HCl (32 %) solution was mixed into each ashed sample and extraction was done using filter paper. Micronutrients and cation concentrations of the extract was measured with a Varian ICP-OES optical emission spectrometer. N content was determined through combustion in a Leco N-analyser on oven dried foliage material.

Heavy metal concentrations for foliar samples were determined according to a similar procedure; after ashing and extraction the aliquot was transferred to a 20 ml ICP tube and analysed using a Varian ICP-OES optical emission spectrometer. Heavy metal content was determined according to the element's wavelength (Section 3.3.3).

Foliar nutrient concentrations at 4 and 8 months after establishment were assessed according to critical levels and the nutrient ratio technique. The critical levels assessment was based on values determined by Dell et al. (1995) and values were used to establish whether foliar nutrient concentrations were in deficient or adequate concentrations. The nutrient ratio technique was used to determine foliar macro and micronutrient concentrations relative to nitrogen. Values were obtained by dividing individual nutrient concentrations by nitrogen and comparing the value produced by "optimal values" determined by Linder (1995). Concentrations were then defined as being deficient or adequate at 4 and 8 months of age.

3.5.6 Growth measurements

Height growth measurements were made using height rods, calibrated in intervals of 10 cm. First measurements were made on the 17th February 2014 at 4 months of

age to the nearest first decimal. Second height measurements were made on the 9th June 2014 at age 8 months to the nearest first decimal.

3.5.7 DBH measurements and Biomass Index

Diameter-at-breast height measurements were taken for each tree on the 9th June 2014 at 8 months after site establishment. Measurements were taken at 1.3 m from the base of each tree with the use of callipers for consistency. DBH values of less than 0.5 cm were defined as being "under DBH" and were included in the analyses as a value of 0.5. A total of 213 trees, or 11.8% of the trial, was defined as "under DBH".

Biomass index was used as a substitute for calculating volume, due to the young age of the trial. A Biomass Index based on the methodology by Donald et al. (1987) was used. The BI incorporated diameter and height measurements into the function and were sampled according to the procedures outlined earlier and in Section 3.5.6. The mean BI per plot was statistically analysed and results are discussed in Chapter 4. Equation 3, determined by Donald et al. (1987), was used to determine a biomass index for each individual tree and the mean BI per plot was substituted into Equation 4 to determine the growth response. The growth response, Equation 4, was adapted from Rolando et al. (2007). Mean BI was calculated for each treatment combination for the trial and subsequently the treatment response was then determined and expressed as a percentage of the growth increase from treatments relative to the control treatment.

Equation 3: $Biomass\ Index = {D_{1.3}}^2\ x\ tree\ height\ (m)$

Where: $D_{1.3}$ = Diameter measured at 1.3 m from the base of the tree in centimetres.

Equation 4: $Treatment\ response = \frac{(treatment\ value - control\ value)}{\text{control value}}\ x\ 100$

3.5.8 Statistical analysis

The effects of wood ash application rate and fertiliser variety on *Eucalyptus grandis x urophylla* growth were analysed using analysis of variance (ANOVA) statistics. A least significant difference ($LSD_{5\%}$) was used to show any significant interaction among treatments if the F statistic was significant for the treatment effect. Treatments with $p<0.05$ were reported as having statistical significance, unless

stated otherwise. Data was initially tested for normality using the Shapiro-Wilk test and Normal Probability plots and homogeneity was tested using the Bartlett's and Levene's Test. Fischer's LSD test was used to compare specific treatment differences for data collected at 4 and 8 months. Data analysis was completed using Statistica 11 software. Regressions were additionally used in the Pilot Study.

3.6 Ethical Considerations

This thesis is compliant with the University of Stellenbosch's policy for responsible research conduct and adheres to the standards set by the Academic Institution. Research was done with integrity and accountability. All individuals and Corporate Sponsors involved in the research are acknowledged in fairness and consideration.

4. Results

Chapter 4 contains the results for the pilot study and data collected and analysed at 4 and 8 months after trial establishment

4.1 Pilot Study

4.1.1 Site soil characteristics

The results of the chemical analysis performed on the soil samples collected during the recognisance survey are shown in Table 4.1. Soil properties were characteristic to that of a coastal sandy soil. The soils had low clay content, low organic matter content and N concentrations of less than 0.05. Individual soil properties will be discussed in the corresponding subsequent sections.

Table 4.1: Basic soil chemical properties for soil samples collected at the experimental site.

	Sample Density	Clay	C	N	Bray II P	K	Ca	Mg	Total Base Cat.	Zn	Mn	Cu	Acid sat.	pH (KCl)
Sample	kg/m^3	%			mg/kg	cmol(+)/kg				mg/kg			%	
1	1470	7	0.6	<0.05	1.36	0.03	1.69	0.19	1.92	0.20	2.72	0.34	5	5.76
2	1480	6	<0.5	<0.05	1.35	0.06	2.11	0.23	2.40	0.14	4.05	0.41	3	6.16
3	1480	5	<0.5	<0.05	1.35	0.02	1.35	0.15	1.52	0.47	2.70	0.47	5	5.55
4	1500	6	<0.5	<0.05	1.33	0.03	1.56	0.18	1.77	0.33	4.00	0.33	4	5.46
5	1510	7	<0.5	<0.05	1.32	0.02	1.01	0.18	1.22	0.20	3.31	0.26	9	4.62
Mean	1488	6.20	-	-	1.34	0.03	1.54	0.19	1.78	0.29	3.36	0.36	5.20	5.51
Std. Error	7.35	0.37	-	-	0.01	0.01	0.18	0.01	0.20	0.06	0.30	0.04	1.02	0.25

4.1.2 Soil buffer capacity

The linear regression graphs for soil #1 and #4 are graphically illustrated in Figures 4.1 and 4.2. Figure 4.1 shows the reaction of increasing wood ash additions and the resultant pH response and Figure 4.2 additions of $CaCO_3$ and the resultant pH response. The methodology required a linear fit and results from the pilot study showed small soil pH increases with increasing additions, even though a sandy soil is known to be poorly buffered. The soil buffer capacities for Figure 4.1 and 4.2 were determined as 74.07 mg $CaCO_3$ (kg soil)$^{-1}$ (pH)$^{-1}$ and 55.56 mg $CaCO_3$ (kg soil)$^{-1}$ (pH)$^{-1}$ respectively.

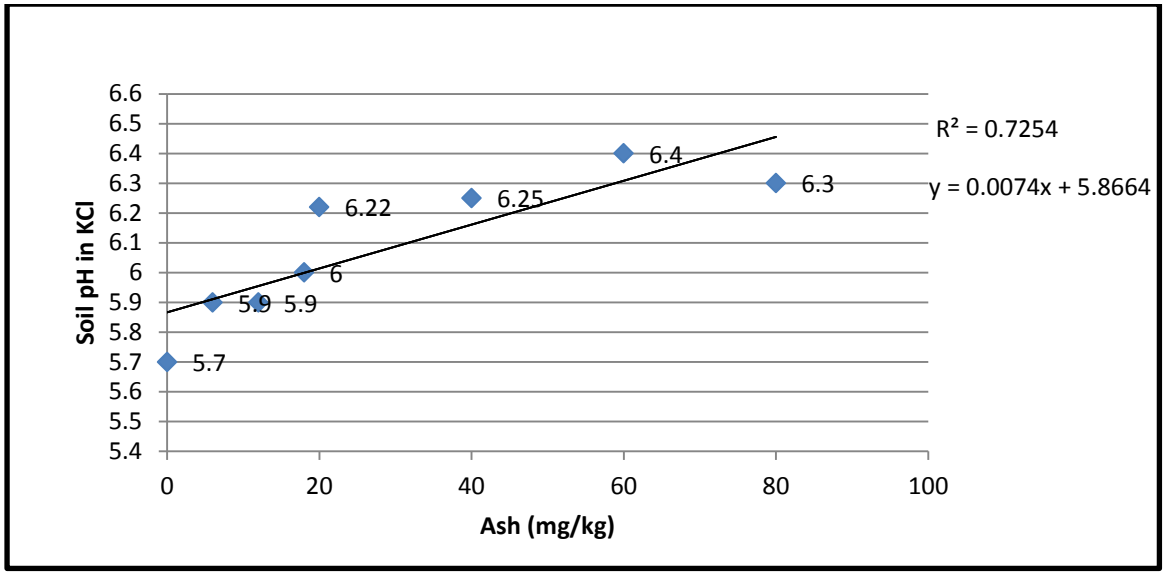

Figure 4.1: Linear Regression for soil sample 1 using wood ash.

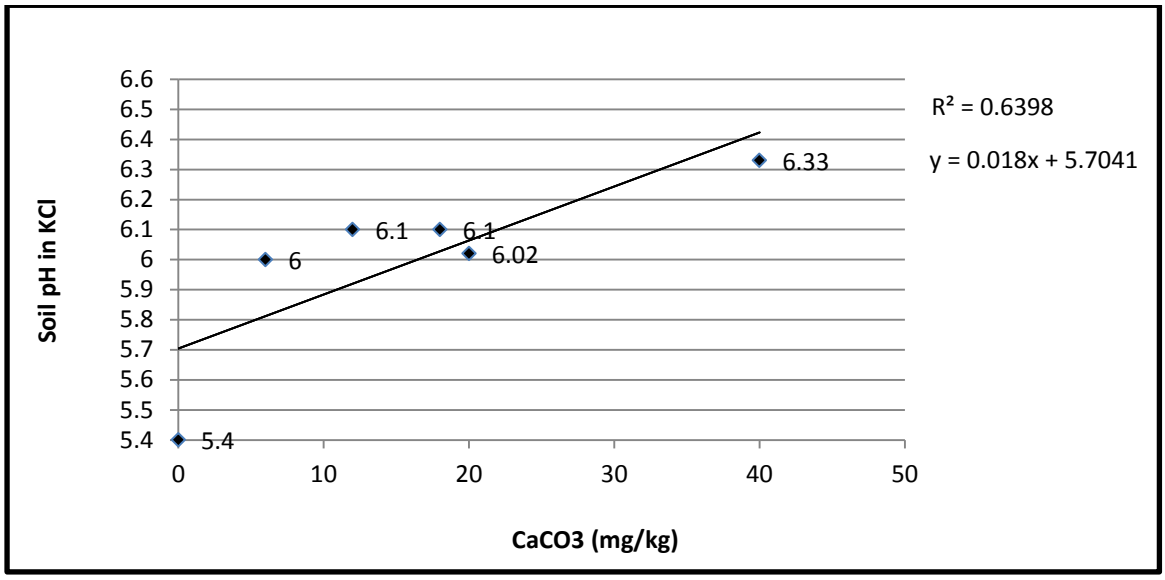

Figure 4.2: Linear Regression for soil sample 4 using pure lime ($CaCO_3$).

4.1.3 Wood ash analysis

This section shows the results obtained from the wood ash analysis conducted in the laboratory prior to trial establishment. Table 4.2 shows the complete elemental breakdown of five wood ash samples. Results showed a mean pH of 12.74 for all 5 samples collected; pH values were high and revealed the ash to contain high hydrous-oxide content. Wood ash sample A3 had a carbon content that was much higher relative to other samples, 17.07%. As expected, all samples had high Ca concentrations, with a mean concentration of 20.44%. The wood ash samples had a

low mean N concentration of 0.04% and revealed the need for a supplementary N source.

Table 4.2: Elemental concentrations for wood ash samples collected

Sample	C	N	P	K	Ca	Mg	Na	Mn	Fe	Cu	Al	Moist.	pH
Units	%						mg/kg					%	
A1	9.30	0.04	0.89	3.72	29.27	3.70	11867	7160	1754	27.9	1742	0.10	12.76
A2	4.57	0.02	0.64	3.78	19.91	2.13	9275	2803	7699	56.7	5419	0.29	11.99
A3	17.07	0.08	1.24	8.23	17.38	17.38	20723	4534	3411	92	2769	0.60	12.14
A4	7.34	0.03	0.86	2.04	27.24	3.30	8171	6110	1254	87	856	1.20	13.40
A5	4.45	0.05	0.36	1.23	8.40	0.91	4046	1320	2117	85	1440	0.50	13.40
Mean	8.55	0.04	0.80	3.80	20.44	5.48	10816	4385	3247	70	2445	0.54	12.74
Std. Error	2.32	0.01	0.15	1.21	3.73	3.01	2779	1063	1169	12	806	0.19	0.30

4.1.4 Wood ash heavy metal analysis

Prior to trial establishment, three wood ash samples were selected for heavy metal analysis to determine Cd, Hg, As, Pb and Cr concentrations. Heavy metal analyses results are presented in Table 4.3. The highest heavy metal concentrations were found for Cr and ranged from 8.93 – 11.57 mg/kg. Two samples showed the presence of Cd and concentrations ranged from 0.70 – 2.10 mg/kg. One sample indicated the presence of Pb, with a concentration of 2.21 mg/kg. Hg and As were not detectable.

Table 4.3: Heavy metal content for three ash samples collected at DukuDuku sawmill.

	Cd	Hg	As	Pb	Cr
Units	mg/kg				
A1	0.696	bd	bd	2.208	11.569
A2	bd	bd	bd	bd	8.929
A3	2.096	bd	bd	bd	10.570

*bd = below detection

4.2 Soil Analysis

This section contains results for soil nutrient and heavy metal concentrations sampled at 4 and 8 months after trial establishment. Soil sampling was not done at time zero (0 months) due to budgetary constraints, and it was initially intended that the control treatments (A_0F_0) be used as the soil conditions at time of establishment. This proved to be problematic as large soil pH variations were found within the trial; results showed that soil pH responded variably to wood ash additions in replication 1 and 2. Potential reasons for the variations are discussed in Chapter 5, Section 5.1.1. Fertiliser control plots were selected for analysis for the whole trial; thus soil analyses were conducted on plots treated purely with wood ash and no fertiliser. Nutrient concentrations were tested for the fixed effects of wood ash application rate, time and the interaction of both variables on nutrient concentrations.

4.2.1 Base cations and effective cation exchange capacity (ECEC)

K^+: Significant treatment differences were found for the fixed effect of time on total soil K^+ concentration. Table 4.4 presents the respective *p-value* and Figure 4.3 the graph for this effect. Mean soil K^+ concentration decreased from 24.04 mg/kg-17.58 mg/kg from 4 to 8 months after trial establishment. In addition, at 4 months standard error was 1.38 mg/kg and 0.0036 mg/kg at 8 months.

Table 4.4: Soil K concentration ANOVA results.

Fixed Effect Test for K (mg/kg)				
Effect	Num. DF	Den. DF	F	p
WA Application	3	20	1.50704	0.243262
Time	1	20	27.36956	0.000040
WA Application*Time	3	20	1.51705	0.240741

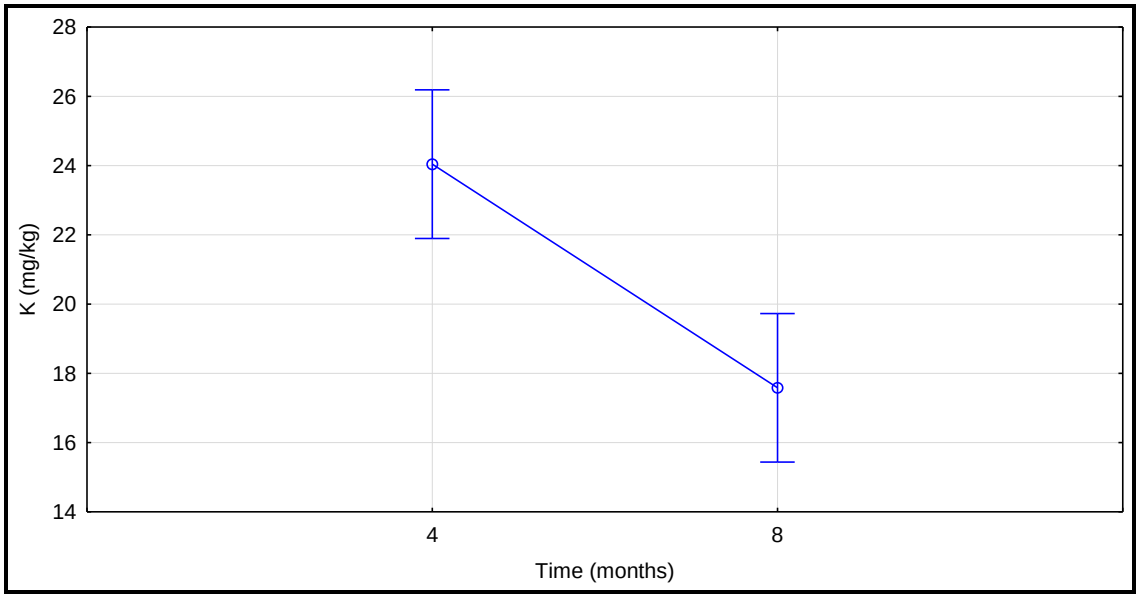

Figure 4.3: Effect of time on mean total soil K^+ concentration after wood ash applications.

Ca^{2+}, Mg^{2+}, Na^+ and exchangeable K^+: The decrease in exchangeable base cations found at 4 to 8 months after trial establishment was expected. Exchangeable Ca^{2+} increased significantly from 4 to 8 months and at the same time exchangeable K^+ and Mg^{2+} decreased; this was anticipated as wood ash predominantly contains Ca. The Ca additions from the wood ash likely displaced the K^+ and Mg^{2+} from the exchange sites soil. K^+ is expressed in cmol(+)/kg, as it refers to the exchangeable K^+. Refer to Table 4.5 for mean and standard error values for base cations and ECEC at 4 and 8 months after trial establishment.

Table 4.5: Mean and standard error of mean concentrations for base cations and ECEC at 4 and 8 months after establishment.

	K	Ca	Mg	Na	ECEC
			cmol(+)/kg		
4 months					
Mean	0.062	1.991	0.498	0.054	2.788
Std. Error	0.002	0.209	0.020	0.002	0.223
8 months					
Mean	0.045	1.182	0.322	0.037	1.838
Std. Error	0.002	0.065	0.011	0.001	0.063

Results indicated significant treatment differences for the fixed effect of time on mean Ca^{2+}, Mg^{2+}, Na^{+} and exchangeable K^{+} cation concentrations in units of centimoles of positive charge per kilogram of soil.

Mean Ca^{2+} concentration was significantly higher at 4 months after trial establishment and showed a decrease by 8 months, i.e. from 2.00 cmol(+)/kg to 1.18 cmol(+)/kg. Over the 4 months mean Ca^{2+} concentration decreased by 0.81 cmol(+)/kg, this decrease showed the temporary effect of wood ash additions on soil Ca^{2+} concentrations in the topsoil. Results showed a mean Mg^{2+} soil concentration of 0.50 cmol(+)/kg at 4 months after trial establishment, and a value of 0.32 cmol(+)/kg at 8 months. In a period of 4 months between measurements the mean Mg^{2+} concentration decreased by 0.18 cmol(+)/kg. Mean Na^{+} concentration was greatest at 4 months after trial establishment and less at 8 months, 0.05 cmol(+)/kg and 0.04 cmol(+)/kg respectively. Mean Na^{+} concentration decreased by 0.02 cmol(+)/kg in a time of 4 months. Although statistically significant, mean K^{+} cation concentration at 4 months and 8 months after trial establishment did not vary considerably. At 4 months mean concentration was 0.06 cmol(+)/kg and at 8months slightly less, with a value of 0.05 cmol(+)/kg. Mean K^{+} concentration decreased with 0.02 cmol(+)/kg for duration of 4 months. Figure 4.5 – 4.8 graphically illustrates the effect of time on Ca^{2+}, Mg^{2+}, Na^{2+} and K^{+} cation soil concentrations after various wood ash treatments. The respective *p-values* for individual cation concentrations are shown in Table 4.6.

Table 4.6: Soil Ca, Mg, Na and K cation concentration ANOVA results.

Fixed Effect Test for Ca (cmol(+)/kg)					
Effect	SS	Df	MS	F	p
WA Application	1.2640	3	0.4213	0.6933	0.561545
Time	7.8570	1	7.8570	12.9296	0.000879
WA Application*Time	0.8527	3	0.2842	0.4677	0.706428
Fixed Effect Test for Mg (cmol(+)/kg)					
Effect	Num. DF	Den. DF	F	p	
WA Application	0.021623	3	0.007208	1.141	0.344397
Time	0.372769	1	0.372769	58.986	0.000000
WA Application*Time	0.005123	3	0.001708	0.270	0.846487
Fixed Effect Test for Na (cmol(+)/kg)					
Effect	Num. DF	Den. DF	F	p	
WA Application	0.000173	3	0.000058	1.010	0.398478
Time	0.003502	1	0.003502	61.350	0.000000
WA Application*Time	0.000040	3	0.000013	0.231	0.874149
Fixed Effect Test for K (cmol(+)/kg)					
Effect	Num. DF	Den. DF	F	p	
WA Application	0.001000	3	0.000333	1.9231	0.141342
Time	0.003333	1	0.003333	19.2308	0.000082
WA Application*Time	0.000600	3	0.000200	1.1538	0.339289

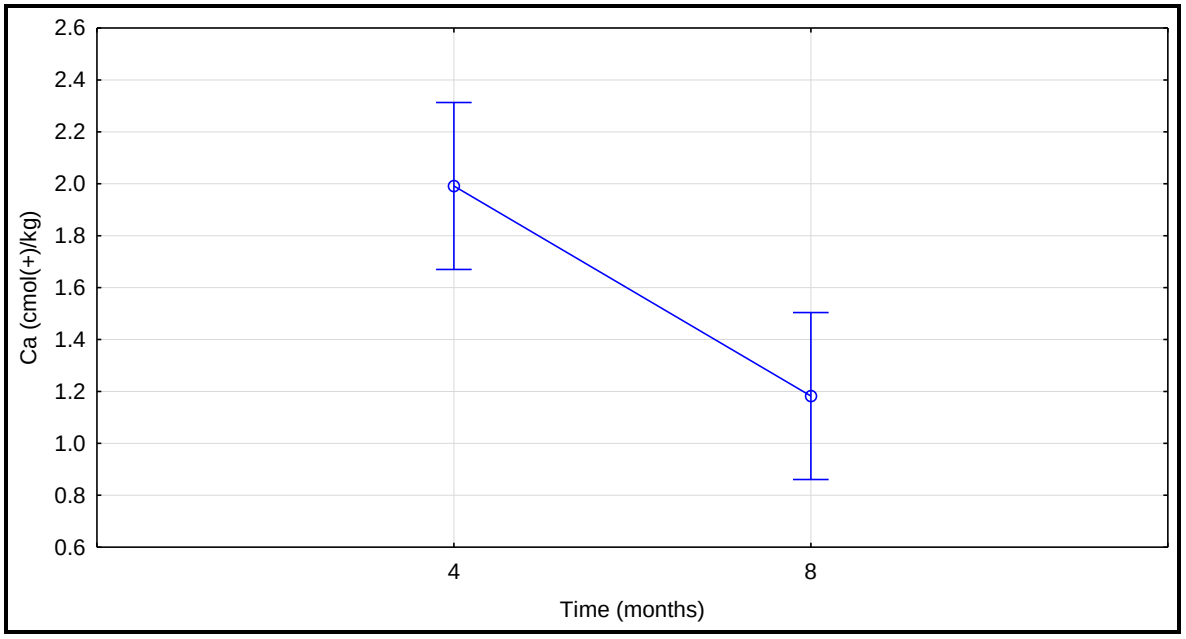

Figure 4.4: Effect of time on mean soil Ca^{2+} concentration after fixed wood ash applications.

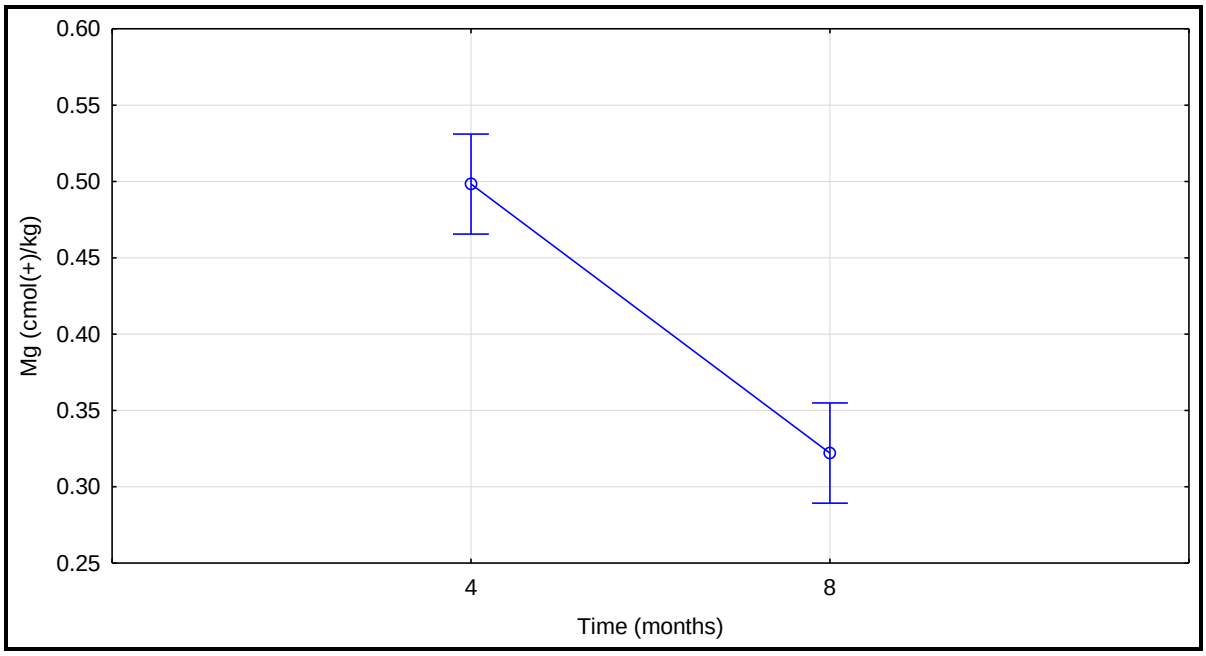

Figure 4.5 Effect of time on mean soil Mg^{2+} concentration after fixed wood ash applications.

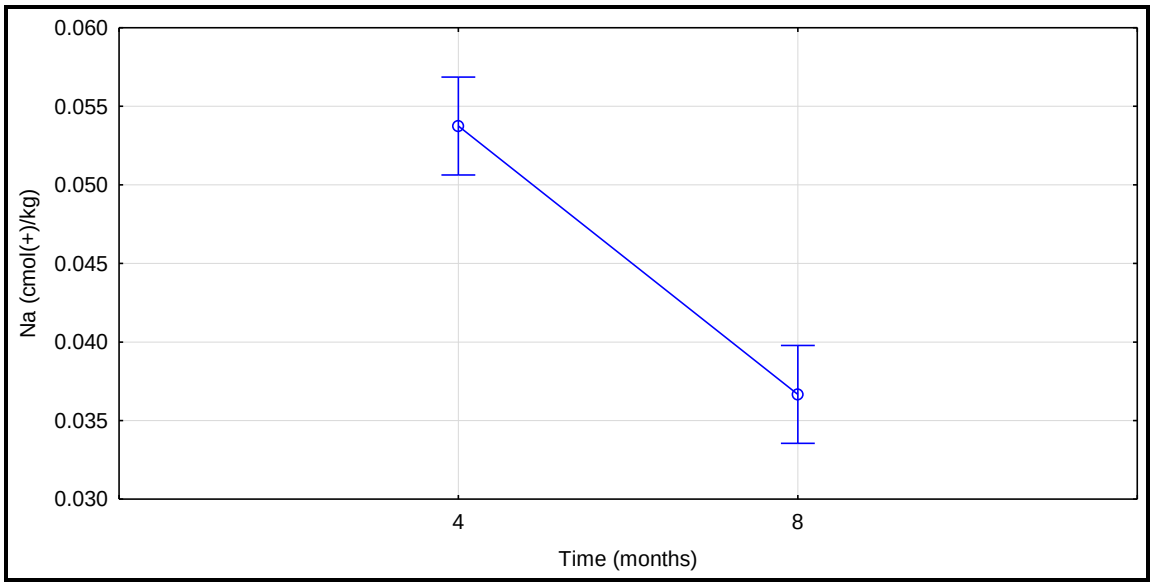

Figure 4.6: Effect of time on mean soil Na^+ concentration after fixed wood ash applications.

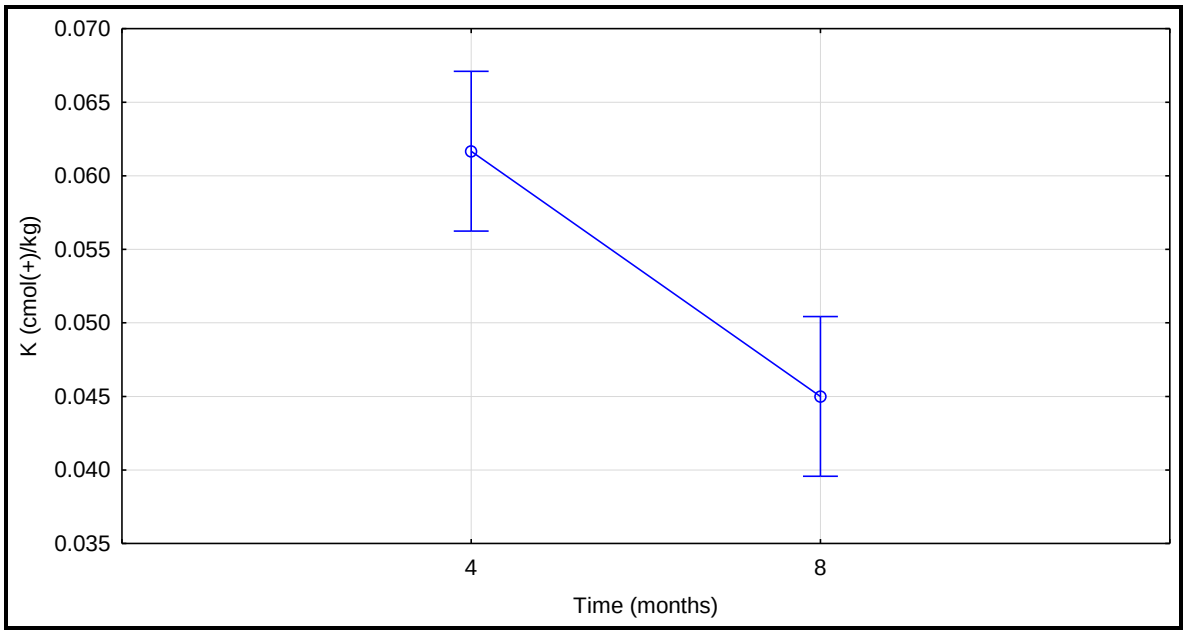

Figure 4.7 Effect of time on exchangeable mean soil K^+ concentration after fixed wood ash applications

ECEC: Results showed significant treatment differences for the effect of time on the mean ECEC of the trial at 4 and 8 months after establishment. This is presented in Table 4.7. The mean ECEC was significantly greater at 4 months and less at 8 months, 2.79 cmol(+)/kg and 1.84 cmol(+)/kg respectively. Although the WA Application*Time interaction was not statistically significant, it was interesting to note

that the greatest mean ECEC of 3.19 cmol(+)/kg was found for plots treated with the highest ash application (1200 kg/ha) at 4 months of age. This showed the initial increase resultant from wood ash applications on soil is greater at the initial period after application and gradually decreases as time progresses. The effect of time on mean ECEC is shown in Figure 4.9.

Table 4.7: ECEC ANOVA results.

		Fixed Effect Test for ECEC (cmol (+)/kg)			
Effect	SS	Df	MS	F	p
WA Application	1.3126	3	0.4375	0.7528	0.527237
Time	10.8205	1	10.8205	18.6179	0.000102
WA Application*Time	0.9288	3	0.3096	0.5327	0.662498

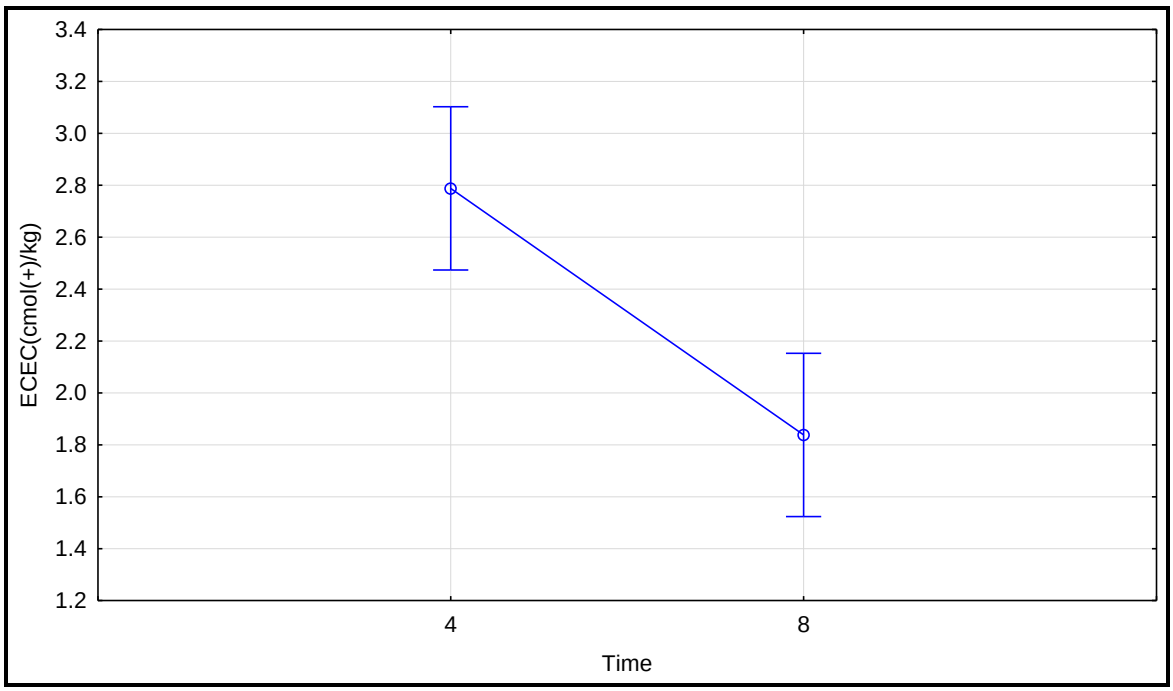

Figure 4.8: Effect of time on mean ECEC after fixed wood ash applications.

4.2.2 Soil pH

Significant treatment differences were found for the effect of time on soil pH across all replications, shown in Table 4.8. Figure 4.10 graphically illustrates the fixed effect of time on soil pH after wood ash applications. Mean soil pH decreased from 6.1 to 5.7 units from 4 to 8 months after trial establishment, thus in a period of 4 months the pH decreased with 0.4 units on average.

Replications 1 and 2 showed unusually high soil pH values for wood ash treatments A_0 to A_3, and pH values were greater relative to other replications. During the reconnaissance survey, wide soil sampling and the subsequent analyses did not reveal high soil pH variability. Only during extensive sampling and analyses throughout the data collection and analysis period did the results reveal high soil pH variability for plots located close to the road. However, replications 3 to 6 did not seem to be affected by possible additions of a liming material. Even though there was some variability, it was found that the soil pH did increase for the experimental period. The liming effect induced by the wood ash in the topsoil horizon of a sandy soil is transitory and decreases several months after application.

As stated at the start of the section; soil measurements at time zero (0 months) were not taken, due to budget constraints and the initial intention of using the control plots as time zero measurements. The high soil pH variability found for the trial, more specifically in replication 1 and 2, brought about the interpretation of the actual mean and standard error values presented in Table 4.9. The soil pH difference for replications 1 and 2, at 4 months, for wood ash treatments of 1200 kg/ha and 0 kg/ha was 1.2 and 0 units. Similarly, at 8 months, results showed soil pH differences of 0.5 and 0 soil pH units for both replications. This showed that soil pH did increase in some instances with increasing ash applications. Soil pH values for replication 2 remained nearly similar at both age intervals. At 8 months after establishment, across all replications, the soil pH values ranged from 5.6 – 6.0 for application rates of 1200 kg/ha and from 5.5 – 5.9 for applications of 0 kg/ha. Soil pH for ash applications of 300 and 600 kg/ha ranged from 5.2 – 6.0 and 5.5 – 6.2 respectively.

Table 4.8: Soil pH ANOVA results at 4 and 8 months after establishment.

	Fixed Effect Test for soil pH			
Replication 1 -6				
Effect	Num. DF	Den. DF	F	p
WA Application	3	20	0.320017	0.810785
Time	1	20	6.130281	0.022351
WA Application*Time	3	20	0.283250	0.836872

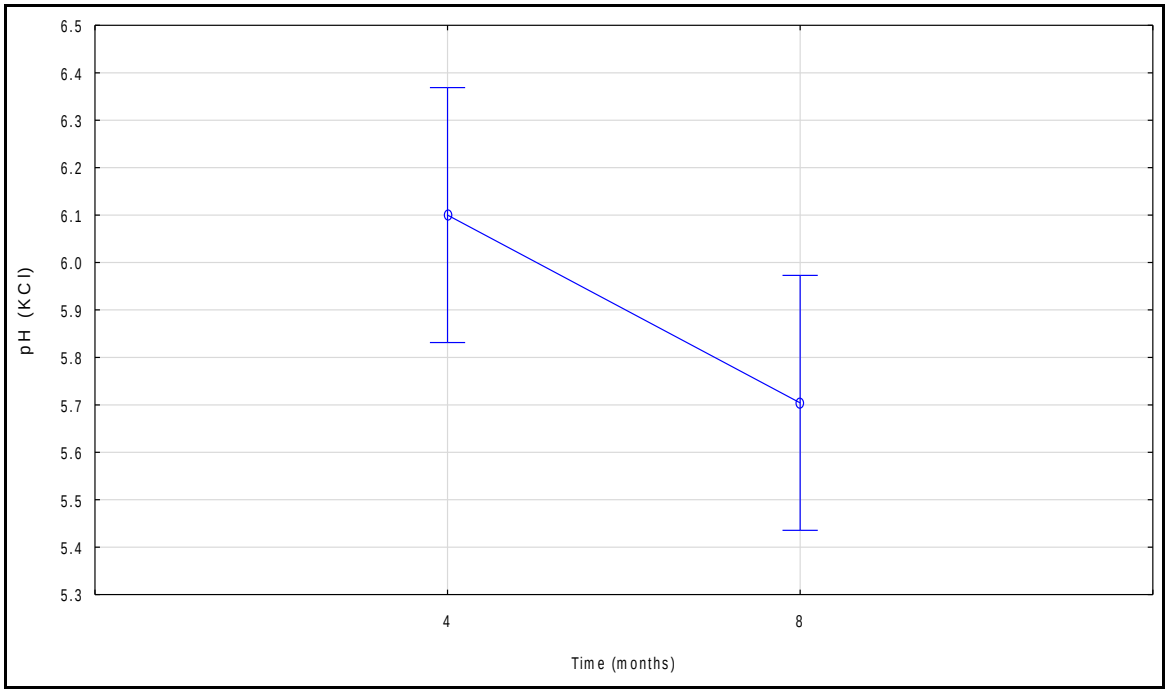

Figure 4.9: Effect of increasing wood ash applications on mean soil pH.

Table 4.9: Mean and standard error of mean and soil pH values with corresponding wood ash application rate at 4 and 8 months after trial establishment.

Application rate	Mean	Std. Error
A0	5.95	0.18
A1	5.94	0.20
A2	5.65	0.15
A3	5.91	0.17

4.2.3 Nutrient levels

Individual nutrients were tested for normality using normal probability curves by means of plotting expected over residual values. The normality assumption is confirmed for all variables, unless stated otherwise. Results indicated no statistical significance for the effect of wood ash application rate on any nutrient concentrations, but the effect of time was significant for all nutrients. In addition, the effect for the interaction of wood ash application and time was not statistically significant for all nutrients. Refer to Appendix 3A and 4A for soil analysis results at 4

and 8 months after trial establishment. Results for soil carbon content, P and K are discussed below:

C%: Results indicated significant treatment differences for the effect of time on soil carbon content and the respective *p-value* is presented in Table 4.10. At 4 months after trial establishment mean soil carbon content was 0.39 %, with a standard deviation of 0.03 %. At 8 months after trial establishment, mean soil carbon content decreased to 0.19 % with a standard deviation of 0.01 %. The greatest mean C content was found for plots treated with the 300 kg/ha wood ash at 4 months after trial establishment and the lowest for plots treated with 0 and 300 kg/ha at 8 months, 0.18 % and 0.17 % respectively. The effect for time on mean C content is graphically illustrated in Figure 4.11.

Table 4.10: Soil carbon content ANOVA results.

Fixed Effect Test for C%					
Effect	SS	df	MS	F	p
Intercept	3.967500	1	3.967500	355.4577	0.000000
Time	0.492075	1	0.492075	44.0862	0.000000
WA Application	0.025617	3	0.008539	0.7650	0.520400
Time*WA Application	0.040942	3	0.013647	1.2227	0.314004
Error	0.446467	40	0.011162		

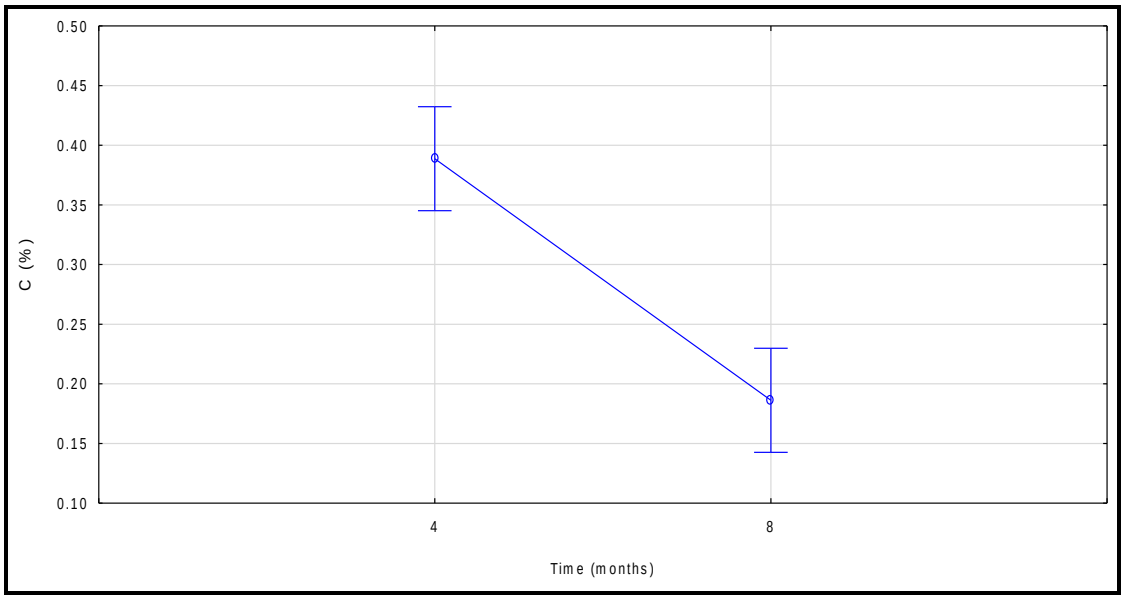

Figure 4.10: Effect of time on mean soil carbon content after wood ash applications.

P: Results showed significant treatment differences for the effect of time on soil P concentration and the respective *p-value* is presented in Table 4.11. Mean soil P concentration was highest at 4 months after trial establishment and significantly less at 8 months, 6.58 mg/kg and 4.00 mg/kg respectively. This is graphically illustrated in Figure 4.12. In addition, standard deviations were 2.47 mg/kg at 4 months and 1.14 mg/kg at 8 months.

Table 4.11: P Bray II concentration ANOVA results.

Fixed Effect Test for P Bray II (mg/kg)				
Effect	Num. DF	Den. DF	F	p
WA Application	3	20	0.70815	0.558402
Time	1	20	20.62232	0.000199
WA Application*Time	3	20	0.53648	0.662654

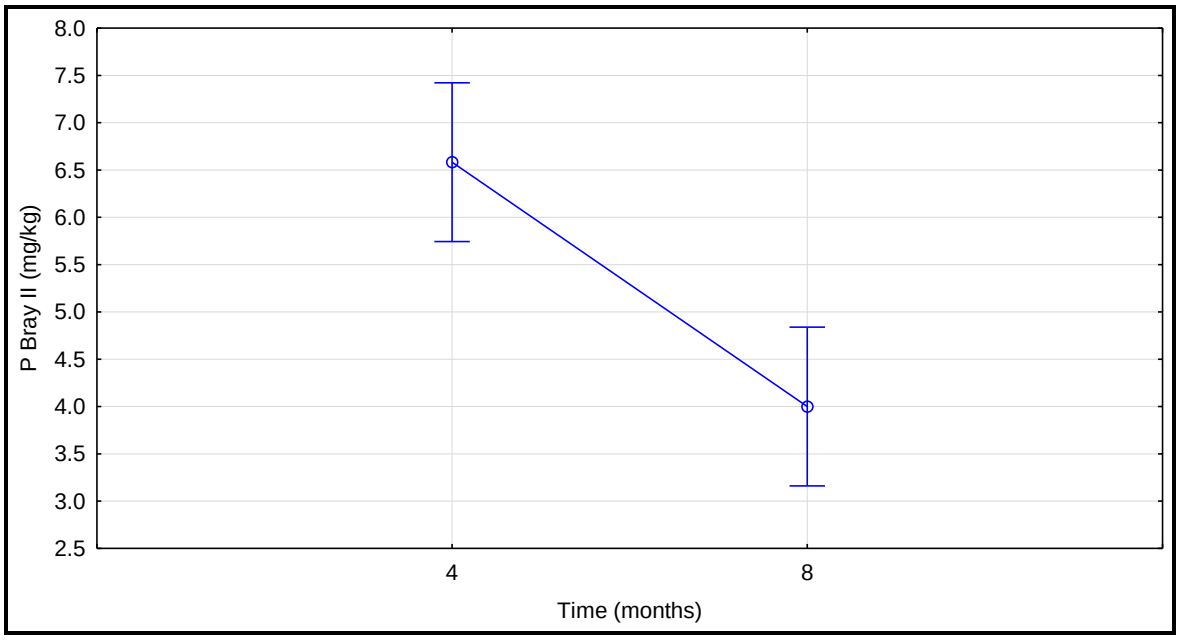

Figure 4.11: Effect of time on mean soil P Bray II concentration after wood ash applications.

4.2.4 Heavy metals

Results indicated significant treatment differences ($p<0.05$) for the fixed effect of time on soil heavy metal concentrations. Table 4.12 presents the ANOVA results for the effect of increasing wood ash applications on individual elements, significant effects are highlighted in red. Heavy metal concentrations were assessed according to

South African guidelines determined by Herselman (2007). Refer to Appendix 5A heavy metal analysis results and the permissible levels determined by Herselman (2007). At 4 and 8 months after trial establishment cadmium was nearly undetectable according to the analysis method described in Section 3.5.3, maximum concentrations of 0.01 mg/kg were found. Refer to Table 4.13 for a summary of mean and standard error of mean heavy metal concentrations. Mercury, chromium and lead concentrations were well under the permissible levels as shown in Appendix 5A.

Table 4.12: Soil heavy metal concentration ANOVA results.

Univariate Tests of Significance for:					
Cd (mg/kg)					
Effect	SS	Df	MS	F	p
Intercept	0.000675	1	0.000675	67.50000	0.000000
Time	0.000675	1	0.000675	67.50000	0.000000
WA Application	0.000025	3	0.000008	0.83333	0.483509
WA application*Time	0.000025	3	0.000008	0.83333	0.483509
Error	0.000400	40	0.000010		
Hg (mg/kg)					
Effect	SS	Df	MS	F	p
Intercept	0.017633	1	0.017633	97.06422	0.000000
Time	0.004800	1	0.004800	26.42202	0.000008
WA Application	0.000433	3	0.000144	0.79511	0.503874
WA application*Time	0.000467	3	0.000156	0.85627	0.471628
Error	0.007267	40	0.000182		
Cr (mg/kg)					
Effect	SS	Df	MS	F	p
Intercept	1103.521	1	1103.521	294.9076	0.000000
Time	319.559	1	319.559	85.3996	0.000000
WA Application	3.124	3	1.041	0.2783	0.840722
WA application*Time	0.692	3	0.231	0.0617	0.979673
Error	149.677	40	3.742		
Pb (mg/kg)					
Effect	SS	Df	MS	F	p
Intercept	279.2228	1	279.2228	353.1878	0.000000
Time	100.4855	1	100.4855	127.1037	0.000000
WA Application	1.3880	3	0.4627	0.5852	0.628200
WA application*Time	0.3721	3	0.1240	0.1569	0.924632
Error	31.6232	40	0.7906		

Table 4.13: Mean and standard error of mean concentrations for heavy metals at 4 and 8 months after establishment.

	Cd	Hg	Cr	Pb
	cmol(+)/kg			
4 months				
Mean	0.000	0.009	2.215	0.965
Std. Error	0.000	0.002	0.114	0.060
8 months				
Mean	0.008	0.029	7.375	3.859
Std. Error	0.004	0.003	0.515	0.238

Mean heavy metal concentration for Hg was highest at 8 months and less at 4 months after trial establishment, 0.03 mg/kg and 0.01 mg/kg respectively. Similar to mercury; mean chromium concentration was highest at 8 months after trial establishment and significantly less at 4 months, 7.38 mg/kg and 2.22 mg/kg. Mean lead concentration was highest at 8 months, with a concentration of 3.86 mg/kg, and less at 4 months after trial establishment with a concentration of 0.97 mg/kg. Figures 4.13-4.16 graphically illustrate the results discussed earlier.

Results showed soil heavy metal concentrations increased from 4 to 8 months after trial establishment. This could possibly be attributed to the significant decrease in soil pH shown in Figure 4.10 and this will be discussed in Chapter 5.

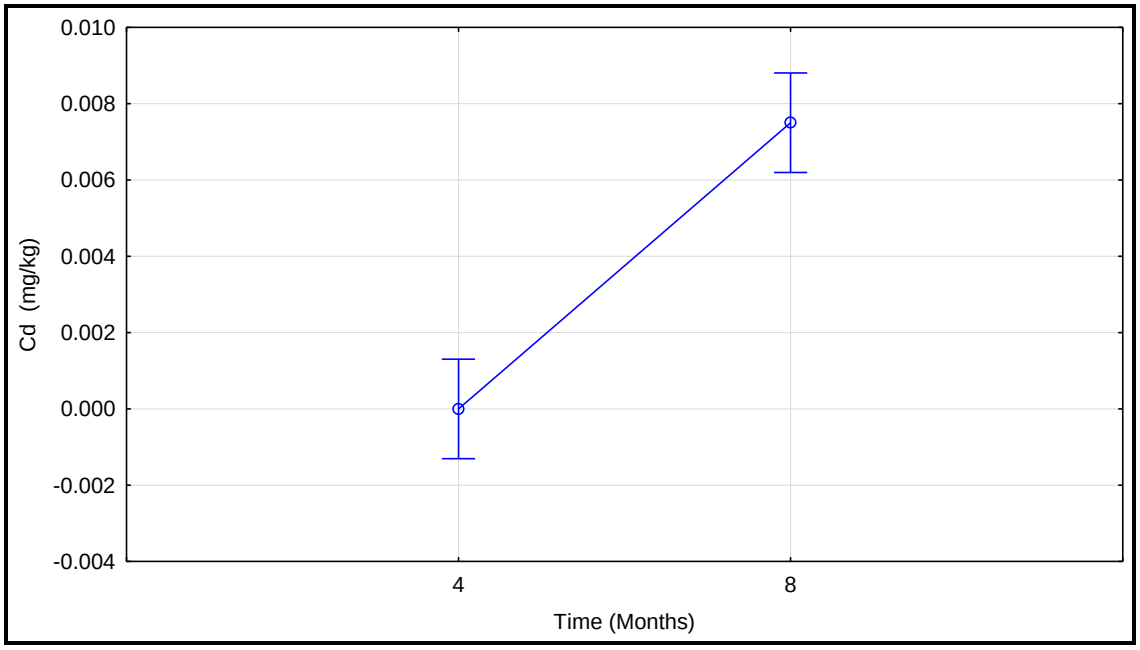

Figure 4.12: Significant effect of time on Cd concentration after fixed wood ash applications of 0, 300, 600 and 1200 kg/ ha were made.

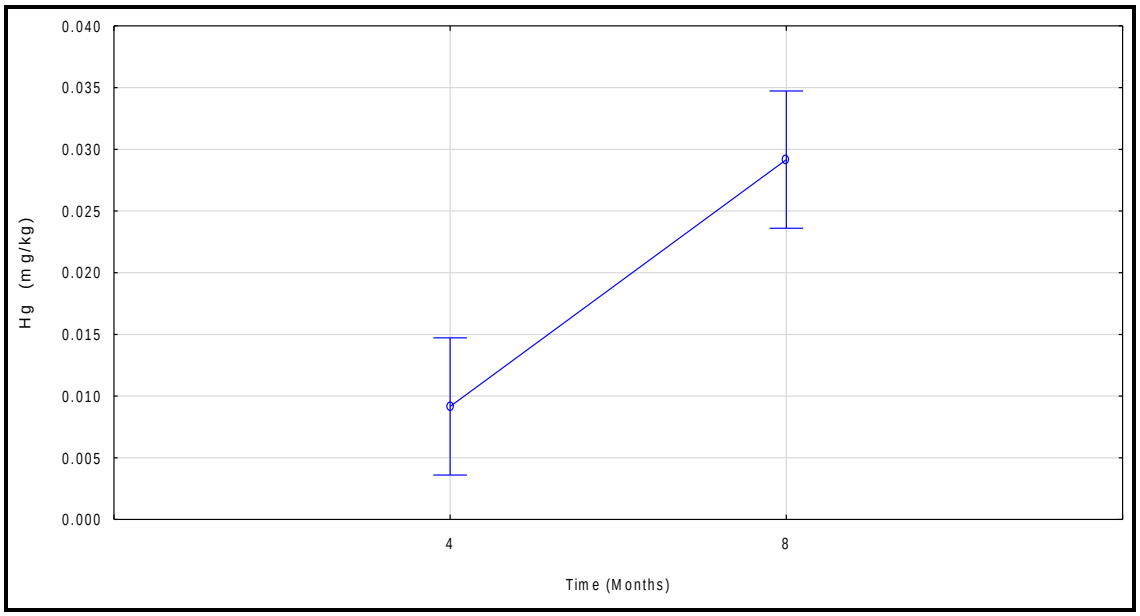

Figure 4.13: Significant effect of time on Hg concentration after fixed wood ash applications of 0, 300, 600 and 1200 kg/ ha were made.

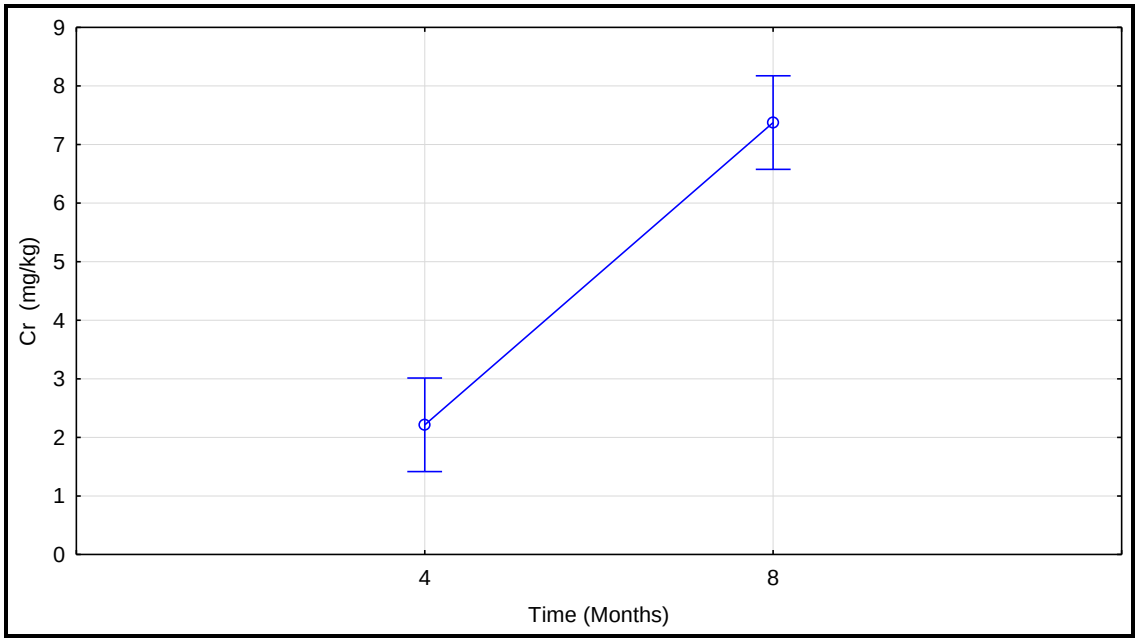

Figure 4.14: Significant effect of time on Cr concentration after fixed wood ash applications of 0, 300, 600 and 1200 kg/ ha were made.

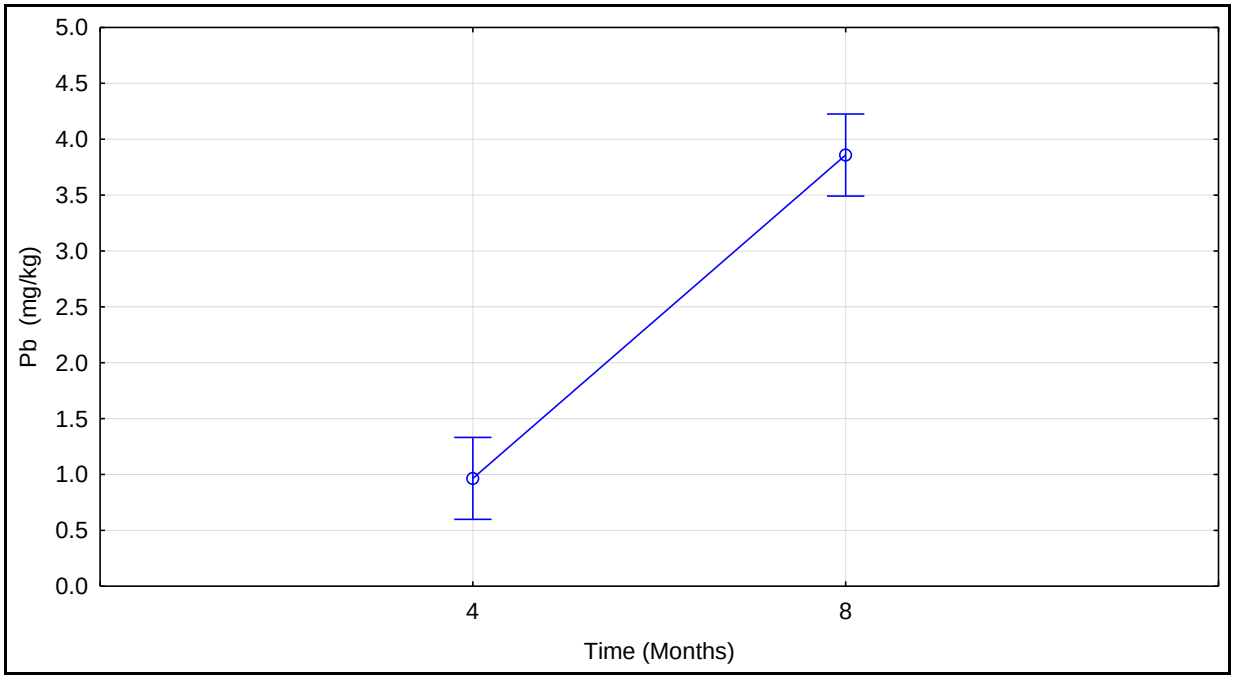

Figure 4.15: Significant effect of time on Pb concentration after fixed wood ash applications of 0, 300, 600 and 1200 kg/ ha were made

4.3 Mortality

Two survival counts were done at 126 and 230 days after trial establishment, at 4 and 8 months of age respectively. At 4 months the trial had an initial mortality of 6.1

%. At 8 months after trial establishment the trial had a final average tree mortality of 4.8% and was less than the 6.1% initially reported at 4 months. The decreased trial mortality reported at 8 months was attributed to seedlings initially marked as dead being sheltered and hidden by coppice re-sprouting from stumps leftover from the previous rotation. As a result of the false mortality count at 4 months of age, the data was excluded from the analyses.

Results showed significant treatment differences for the effect of the fertiliser type used in the experiment at 8 months of age. Table 4.14 shows the statistical significance of the main effects for fertiliser and wood ash on mortality recorded at 8 months of age. Statistica software did not generate an interaction for the effects of wood ash applications and fertiliser; this was likely due to the small dataset as a result of excluding 4 month data from the analysis. In declining order, results showed the highest mean mortality for F_0, F_{CRF} and lastly F_{CV} treated plots, the values being, 10, 9 and 6 trees per treatment respectively. A_0 treated plots had the highest mean mortality and A_2 treated plots the lowest, 10 and 6 trees per treatment respectively. Table 4.15 presents the total seedling mortality for different wood ash and fertiliser treatment combinations 8 months of age and respective percentages of total trial mortality for each treatment. Several months before final mortality count, a coppice reduction was done and revealed better seedling survival figures. A total of 22 trees initially reported as dead were counted at 8 months of age and A_1F_0 and A_1F_{CRF} treated plots had the highest mortality count, 12 and 11 trees per treatment respectively.

Table 4.14: Mortality ANOVA results 8 months of age.

Effect	SS	df	MS	F	p
Intercept	630.7500	1	630.7500	129.7543	0.000001
WA Application	14.9167	3	4.9722	0.51437	0.683748
Fertiliser	48.5000	2	24.2500	4.9886	0.034838
Error (WA Application)	77.3333	8	9.6667		
Error (Fertiliser)	43.7500	9	4.8611		

Table 4.15: Percentage mortality of total mortality for each treatment across all replicates at 8 months after trial establishment for different wood ash and fertiliser combinations.

WA Application (kg/ha)	Fertiliser	Percentage of total trial mortality (%).	Mortality at 8 months (No. of trees).
A_0	F_0	10	9
A_0	F_{CRF}	11	10
A_0	F_{CV}	6	5
A_1	F_0	14	12
A_1	F_{CRF}	13	11
A_1	F_{CV}	3	3
A_2	F_0	10	9
A_2	F_{CRF}	6	5
A_2	F_{CV}	5	4
A_3	F_0	8	7
A_3	F_{CRF}	7	6
A_3	F_{CV}	7	6

4.4 Mean height growth

Mean height growth per plot was used to do a statistical analysis.

Table 4.16 shows the ANOVA test of significance for the effect of wood ash, fertiliser and the interaction of both variables. Results found statistical significance only of the effect for fertiliser and indicated treatment differences within the data. Figure 4.17 graphically illustrates the evidence for treatment differences for the effect of fertiliser using the Least squares means. Figure 4.18 and 4.19, taken at 8 months of age, shows the height growth for plot 33 relative to plot 65. Plot 33 was treated with A_1F_{CV} and plot 65 with A_0F_0. Mean height values for F_0, F_{CV} and F_{CFR} treated plots were 200, 240 and 268 cm respectively. Standard error for all plots treated with either one of the three fertilisers was 7 cm. F_{CRF} treated plots had a greater mean height and proved to be superior to unfertilised and F_{CV} treated plots. Refer to Appendix 6A for a complete statistical breakdown for wood ash and fertiliser treatments.

Table 4.16: ANOVA results for mean heights at 8 months of age.

Univariate Tests of Significance for:					
Effect	SS	Df	MS	F	p
Mean height					
Intercept	4010490	1	4010490	3264.637	0.000000
WA Application	1450	3	483	0.394	0.758089
Fertiliser	55008	2	27504	22.389	0.000000
Fertiliser*WA Application	6393	6	1065	0.867	0.524246
Error	73708	60	1228		

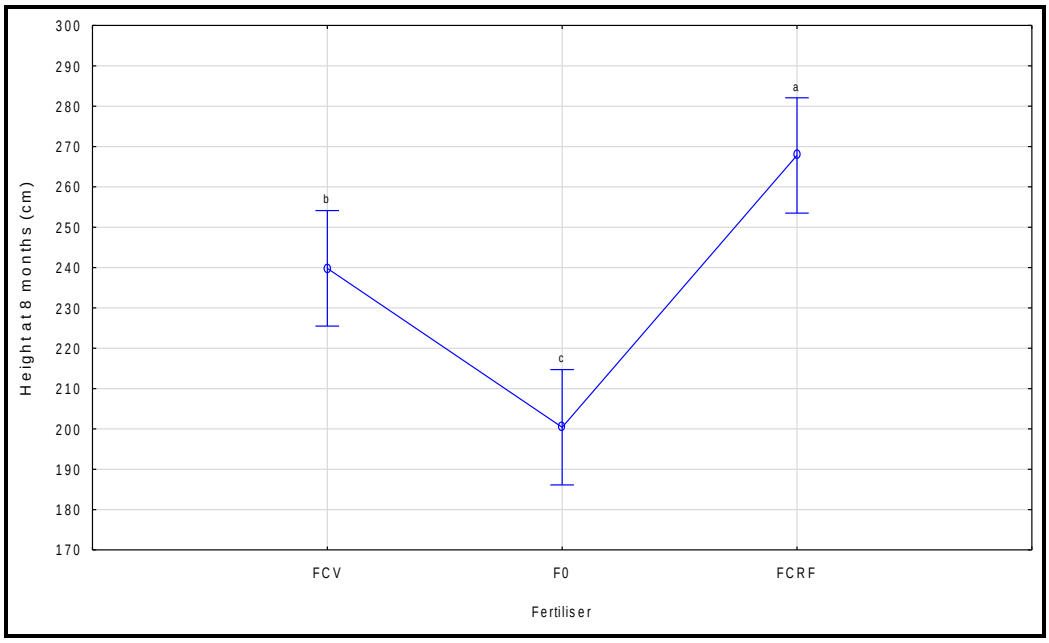

Figure 4.16: Treatment differences and the effect of F_0, F_{CV} and F_{CFR} on mean tree height at 8 months of age

Figure 4.17: Plot 33, treated with A_1F_{CV} at 8 months of age (Pictured: Victor Msane).

Figure 4.18: Plot 65, treated with A_0F_0 at 8 months of age. (Scale: Burnt stump on the left is approximately 1m tall).

4.5 Diameter growth

Mean DBH per plot was used in the data analyses. The Shapiro-Wilk test statistic and expected-over-residual plot was used to show normality (p<0.05). The Shapiro-Wilk test showed p=0.91945 and provided evidence for normality.

Table 4.17 presents the tests of significance for the effects of wood ash, fertiliser and the interaction at 8 months after trial establishment. The effect for fertiliser on DBH showed statistical significance and indicated treatment differences (p<0.05). Mean treatment differences for fertiliser are graphically shown in Figure 4.20.

Table 4.17: ANOVA results for DBH at 8 months of age.

Univariate Tests of Significance for:					
Effect	SS	Df	MS	F	p
DBH at 8 months					
Intercept	212.3723	1	212.3723	1054.519	0.000000
WA Application	0.5495	3	0.1832	0.910	0.441869
Fertiliser	8.5312	2	4.2656	21.181	0.000000
Fertiliser*WA Application	1.6698	6	0.2783	1.382	0.236691
Error	12.0836	60	0.2014		

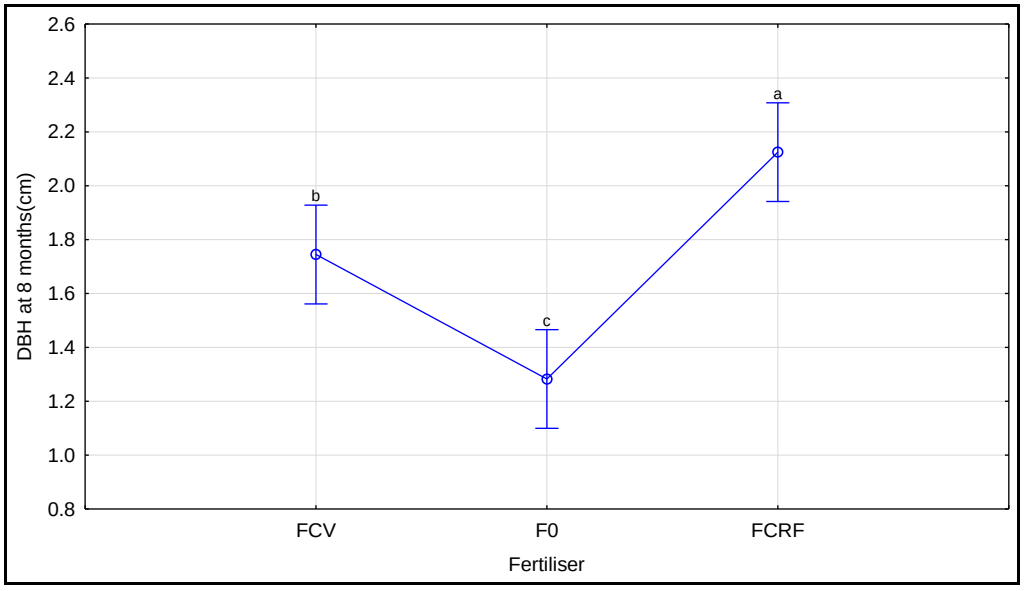

Figure 4.19: Treatment differences and the effect of F_0, F_{CV} and F_{CFR} on tree DBH.

Mean values for F_0, F_{CV} and F_{CRF} treated plots were 1.3 cm, 1.7 cm and 2.12 cm respectively, thus indicating F_{CRF} treated plots had greater diameter increment

relative to other treatments. Refer to Appendix 7A for a statistical breakdown of all fertiliser and wood ash treatments.

4.6 Biomass Index & Biomass Index Growth Response

Results showed no statistically significant interactions (p<0.05) on a 95 % confidence level for the fixed effects of wood ash application, fertiliser and the interaction of both variables on the BI. However, the main effect for wood ash application rate was statistically significant (p<0.1) for a 90 % confidence level. Table 4.18 shows *p-values* for corresponding effects. Figure 4.21 graphically illustrates the effect of wood ash application on mean biomass index.

Table 4.18: ANOVA results for mean biomass index per plot determined at 8 months of age.

Univariate Tests of Significance for:					
Effect	SS	Df	MS	F	p
Tree DBH					
Intercept	16947.94	1	16947.94	25.65044	0.000004
WA Application	1899.83	3	633.28	0.95845	0.418302
Fertiliser	3462.73	2	1731.36	2.62039	0.081089
WA Application*Fertiliser	4409.64	6	734.94	1.11232	0.365998
Error	39643.61	60	660.73		

Results showed the greatest mean biomass index for F_{CRF} treated plots and the poorest for unfertilised plots (F_0), 22.92 and 6.16 respectively. F_{cv} treated plots had an intermediary biomass index of 16.94.

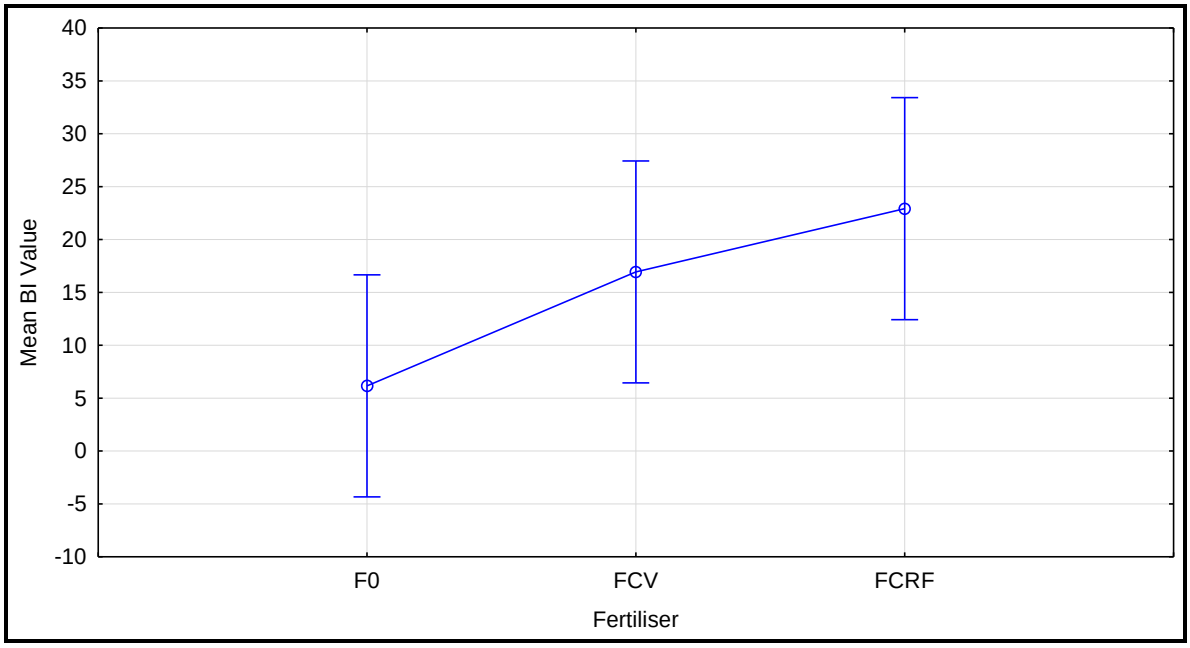

Figure 4.20: Treatment differences and the effect of F_0, F_{CV} and F_{CFR} on mean BI at 8 months of age

The growth response for treatments relative to the control is shown in Table 4.19. Results indicated the poorest growth response from A_2F_0, A_1F_0 and A_3F_0 treatments that received no supplementary nutrition, 13 %, 21 % and 48 % respectively. In addition, the top 3 underachieving treatments showed that the highest wood ash application rate is superior to the remaining treatments that received 300 and 600 kg/ha wood ash in the absence of supplementary nutrition. The overall best growth response was found for plots treated with control released fertiliser (F_{CRF}) and wood ash combinations; the best mean growth response of 683 % was found for A_2F_{CRF} treatments. However A_0F_{CV} treatments were the exception and had a mean growth response of 644 % relative to the control, even though it did not receive any wood ash. This treatment surpassed the remaining conventional fertiliser and wood ash treatments with an exceptional growth response. Figures 4.22 and 4.23 show individual treatment differences for 2 plots, taken at 8 months of age.

Table 4.19: Biomass index growth response for treatment combinations relative to the control treatment at 8 months of age.

Treatment	Mean BI for each Treatment	Response (%)
	Control treatment	
A_0F_0	5.12	0.00
	Treatments	
A_2F_0	5.76	12.55
A_1F_0	6.19	21.09
A_3F_0	7.59	48.33
A_3F_{CV}	8.19	60.14
A_2F_{CV}	9.81	91.85
A_1F_{CV}	11.70	128.75
A_1F_{CRF}	12.84	151.11
A_3F_{CRF}	15.66	206.13
A_0F_{CRF}	23.14	352.36
A_0F_{CV}	38.05	643.91
A_2F_{CRF}	40.05	682.96

Figure 4.21: Plot 66, treated with A_2F_{CRF} at 8 months after trial establishment

Figure 4.22: Plot 68, treated with A_3F_0 at 8months after trial establishment. Seen from plot 67; treated with A_1F_0.

4.7 Foliar Analysis

The laboratory results for foliage samples are discussed here. Twenty four plots, replications 2 and 4, were selected for foliar analysis.

4.7.1 Critical levels at respective ages.

Nutrient concentrations were assessed according to the critical levels method determined by Dell et al. (1995). Table 4.20 shows the chemical analysis for the plots 1 - 24 analysed at 4 months of age and Table 4.21 at 8 months, including critical levels for individual nutrients as determined by Dell et al. (1995). The critical level for Na, determined by Reuters and Robinson (1997), is also shown in both tables. The chemical analysis at 4 months of age, shown in Table 4.20, indicated sub-optimal nutrient concentrations for P, K and Zn. The identified plots were defined as being sub-optimal due to nutrient concentrations occurring between adequate and deficient levels. Refer to Appendix 1A and 2A for the plots identified with sub-optimal nutrient concentrations. At 4 months of age, the highest N concentration was 3.10 % for two plots treated with A_1F_{CRF} and A_2F_{CRF}, but concentrations varied little relative to other treatments as concentrations ranged from 2.34 % - 3.10 %. An A_1F_{CRF} treated plot had the highest P concentration of 0.25 %, and similarly the concentrations varied little between treatments and ranged from 0.14 %–0.25 %. An A_1F_0 control plot had the highest K concentration of 1.23 % and concentrations ranged from 0.86 % – 1.23 % for all treatments.

At 8 months of age, shown in Table 4.21, several plots indicated potentially sub-optimal K concentrations that could constrain growth. Additionally a single plot revealed the possibility of sub-optimal P concentrations. An A_0F_{CRF} treated plot had the highest N concentration and a control plot (A_0F_0) the lowest, 3.15 % and 1.98 % respectively. The analysis revealed an A_1F_{CRF} plot had the highest P concentration, but concentrations ranged between 0.14 % – 0.24 % for all plots analysed. K concentrations ranged between 0.85 % – 1.11 %, with an A_1F_{CRF} treated plot having the highest concentration.

Table 4.20: Foliar nutrient concentrations at 4 months after trial establishment Critical nutrient levels for *Eucalyptus grandis x urophylla* at age 1 and 2 years (Dell et al., 1995).

Plot nr.	N	P	K	Ca	Mg	Na	Mn	Fe	Cu	Zn	B	Treat.
	%					mg/kg						
1	3.05	0.17	0.86	0.6	0.29	2184	233	58	6	13	37	A_2F_{CRF}
2	3.00	0.18	0.95	0.68	0.30	2497	244	65	7	15	37	A_0F_{CV}
3	2.95	0.18	1.19	0.82	0.32	3200	180	67	8	16	38	A_3F_0
4	2.85	0.16	1.07	0.64	0.29	3040	222	58	7	13	36	A_0F_0
5	2.79	0.16	1.07	0.64	0.31	3358	299	69	7	14	37	A_3F_{CV}
6	2.99	0.16	1.07	0.67	0.30	2881	245	58	7	15	38	A_2F_{CV}
7	2.63	0.15	0.99	0.51	0.27	2642	249	55	6	14	38	A_1F_{CV}
8	2.72	0.17	1.10	0.75	0.31	3159	215	59	8	15	40	A_1F_0
9	2.62	0.20	1.20	0.85	0.37	3380	299	64	8	18	44	A_2F_0
10	3.05	0.19	0.99	0.68	0.34	2960	351	71	7	14	41	A_1F_{CRF}
11	2.87	0.19	0.95	0.74	0.32	2990	314	62	7	14	41	A_0F_{CRF}
12	2.83	0.17	1.01	0.65	0.29	2645	293	55	7	13	36	A_3F_{CRF}
13	2.90	0.20	1.01	0.65	0.35	2639	248	59	6	12	38	A_2F_{CRF}
14	2.65	0.16	1.02	0.71	0.36	3065	307	59	7	15	39	A_3F_{CV}
15	2.65	0.14	1.05	0.63	0.29	3056	239	53	7	15	36	A_0F_{CV}
16	2.53	0.14	1.00	0.66	0.30	3264	189	56	8	14	35	A_1F_0
17	2.34	0.20	1.23	0.72	0.31	2931	214	54	8	16	41	A_0F_0
18	2.60	0.17	1.13	0.66	0.31	2860	219	63	7	15	43	A_3F_0
19	2.74	0.18	1.13	0.64	0.31	2941	226	65	7	15	42	A_2F_0
20	2.71	0.20	1.13	0.59	0.31	2720	218	60	7	14	39	A_2F_{CV}
21	3.10	0.25	1.12	0.59	0.35	2759	239	66	6	13	42	A_0F_{CRF}
22	2.58	0.17	1.12	0.53	0.29	3009	276	63	7	15	37	A_1F_{CV}
23	2.82	0.22	1.09	0.51	0.32	2696	225	62	6	13	39	A_3F_{CRF}
24	3.10	0.19	0.99	0.56	0.30	2449	239	61	7	13	38	A_1F_{CRF}
Deficient	0.8-1.1	0.09-0.1	0.2-0.6	n/a	0.02-0.04	n/a	n/a	n/a	n/a	n/a	8.0-12.0	
Adequate	1.8-2.9	0.15-0.26	0.9-1.5	0.21-0.75	0.11-0.36	0.30-0.42	134-2316	41-98	3.5-3.4	13-29	13-30	

*Shaded cells = potentially deficient or imbalanced nutrient concentrations.

Table 4.21: Foliar nutrient concentrations 8 months after trial establishment and Critical nutrient levels for *Eucalyptus grandis x urophylla* at age 1 and 2 years (Dell et al.,1995).

Plot nr.	N	P	K	Ca	Mg	Na	Mn	Fe	Cu	Zn	B	Treatment
	%					mg/kg						
1	3.02	0.18	0.89	0.68	0.29	4018	363	136	5	17	32	A_2F_{CRF}
2	2.52	0.14	0.94	0.69	0.29	4684	324	114	6	16	27	A_0F_{CV}
3	2.23	0.18	0.92	0.77	0.34	4869	274	113	7	17	24	A_3F_0
4	2.19	0.18	0.95	0.68	0.33	5000	331	118	7	16	27	A_0F_0
5	2.78	0.17	0.95	0.69	0.3	4883	378	123	8	17	26	A_3F_{CV}
6	2.97	0.2	1.06	0.65	0.3	4512	283	116	8	18	25	A_2F_{CV}
7	2.93	0.21	1.1	0.65	0.29	4344	318	114	8	18	24	A_1F_{CV}
8	2.32	0.19	1.01	0.69	0.31	4931	274	103	8	16	20	A_1F_0
9	2.33	0.19	0.92	0.73	0.32	4615	327	105	7	15	22	A_2F_0
10	2.93	0.24	1.11	0.71	0.32	4735	409	110	6	18	26	A_1F_{CRF}
11	3.15	0.19	0.87	0.58	0.26	3978	332	97	5	14	26	A_0F_{CRF}
12	2.87	0.21	1.06	0.64	0.29	4403	349	98	5	16	26	A_3F_{CRF}
13	3.01	0.2	1.03	0.68	0.28	4221	281	100	5	16	27	A_2F_{CRF}
14	2.72	0.17	0.89	0.78	0.29	4231	333	110	8	17	24	A_3F_{CV}
15	2.27	0.15	0.94	0.68	0.31	4859	300	109	8	16	22	A_0F_{CV}
16	2.05	0.16	0.85	1.26	0.34	4910	306	151	8	17	22	A_1F_0
17	1.98	0.18	0.88	0.83	0.35	4962	296	112	7	17	21	A_0F_0
18	2.09	0.22	1.08	0.86	0.4	5733	404	111	9	18	23	A_3F_0
19	2.26	0.18	0.97	0.73	0.33	4767	299	108	7	16	24	A_2F_0
20	2.72	0.17	0.97	0.69	0.29	4448	300	112	7	17	25	A_2F_{CV}
21	3.03	0.2	1.02	0.65	0.27	3824	301	113	5	16	28	A_0F_{CRF}
22	2.92	0.2	1.04	0.59	0.27	3951	267	99	5	15	26	A_1F_{CV}
23	2.68	0.17	0.89	0.66	0.27	3820	316	88	7	16	22	A_3F_{CRF}
24	2.98	0.18	0.95	0.68	0.26	3663	290	110	5	16	27	A_1F_{CRF}
Deficient	0.8-1.1	0.09-0.1	0.2-0.6	n/a	0.02-0.04	n/a	n/a	n/a	n/a	n/a	8.0-12.0	
Adequate	1.8-2.9	0.15-0.26	0.9-1.5	0.21-0.75	0.11-0.36	0.30-0.42	134-2316	41-98	3.5-3.4	13-29	13-30	

*Shaded cells = potentially deficient or imbalanced nutrient concentrations.

Individual nutrient concentrations were subjected to an ANOVA in order to determine the statistical significance of the effects for fertiliser, time, wood ash application and the interaction of the above have on nutrient concentrations.

N concentration: The statistical significance for the fixed effects is presented in Table 4.22. Results showed statistical significance for the effect of fertiliser, time,

fertiliser*time interaction and the interaction for wood ash*fertiliser*time. This indicated significant treatment differences.

Table 4.22: Foliar N concentration ANOVA results.

Effect	Num. DF	Den. DF	F	P
WA Application	3	12	0.97241	0.437761
Fertiliser	2	12	32.02734	0.000015
Time	1	12	45.87676	0.000020
WA Application*Fertiliser	6	12	0.94955	0.496477
WA Application*time	3	12	3.16613	0.063925
Fertiliser*time	2	12	38.85108	0.000006
WA Application*Fertiliser*time	6	12	5.88315	0.004580

Mean nitrogen concentrations for F_0, F_{CV} and F_{CRF} treated plots were 2.43 %, 2.74 % and 2.96 % respectively. The effect for time was statistically significant and indicated differences at 4 and 8 months of age. At 4 months of age mean foliar N concentration was 2.79 % and at 8 months 2.62 %.

The effect for fertiliser variety and time is shown in Figure 4.24. Mean N concentrations were highest for F_{CRF} treatments at 4 months of age and slightly less at 8 months, 2.97 % and 2.96 % respectively. The lowest mean N concentrations were found for unfertilised treatments at 4 and 8 months of age, 2.67 % and 2.18 % respectively. The mean N concentration for F_0 plots at 8 months of age was significantly lower than other mean concentrations found for F_{CFR} and F_{CV} treated plots of the same age. Figure 4.25 graphically illustrates the effect of wood ash application, time and fertiliser variety on N concentration and significant treatment differences. The highest mean N concentration of 3.09 % was found for A_0F_{CRF} treated plots at 8 months of age and the lowest mean N concentration of 2.09 % for plots treated with A_0F_0 at 8 months of age.

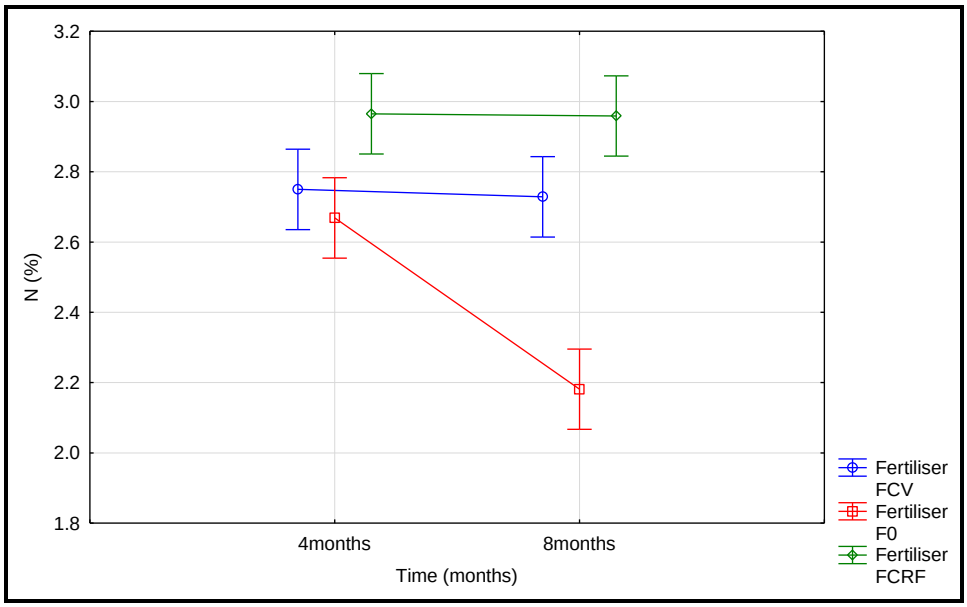

Figure 4.23: Foliar N concentrations for the effect of time and fertiliser variety.

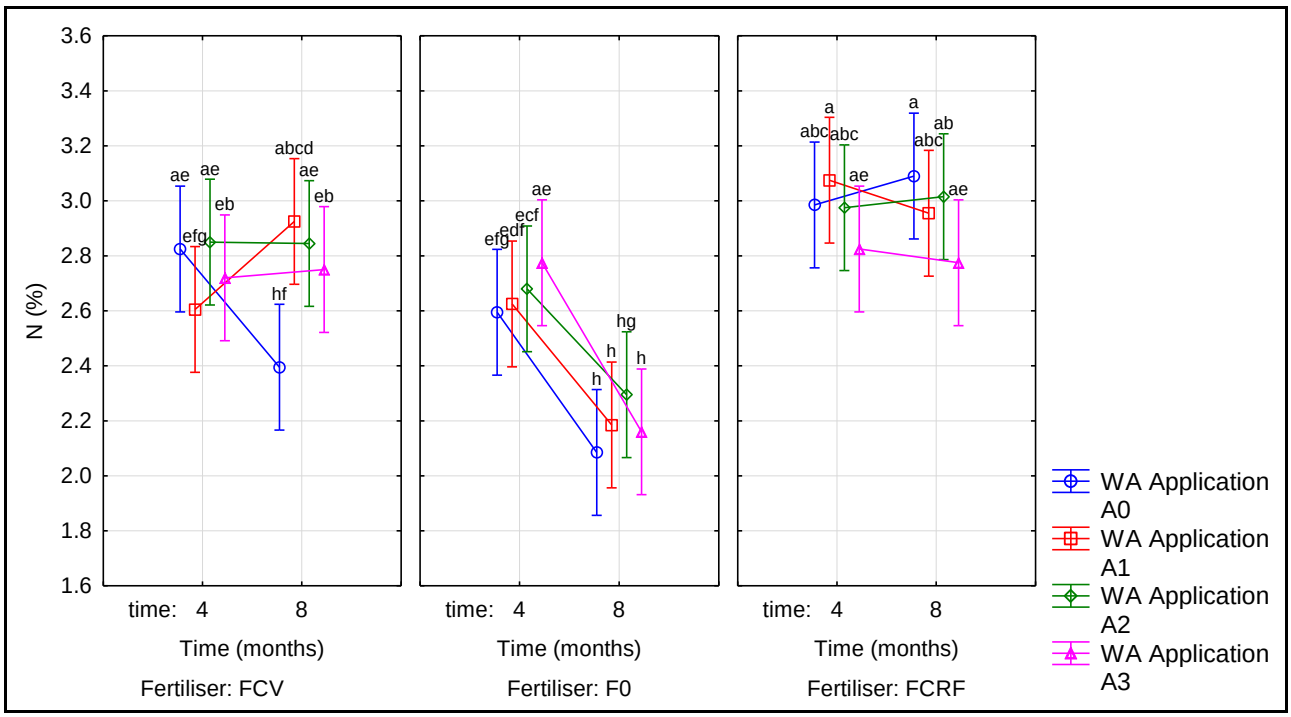

Figure 4.24: Treatment differences and mean foliar N concentrations for the effect of 12 treatment combinations of fertiliser, time and wood ash application rate.

P: Significant treatment differences and corresponding *p-values* are presented in Table 4.23. A significant treatment difference was found for the effect of fertiliser on foliar P concentration and is graphically illustrated in Figure 4.26. F_{CRF} treated plots

had the highest mean P concentration and F_{CV} treated plots the lowest, 0.197 % and 0.171 % respectively.

Table 4.23: Foliar P concentration ANOVA results.

Effect	Num. DF	Den. DF	F	P
WA Application	3	12	0.150725	0.927225
Fertiliser	2	12	5.908696	0.016358
Time	1	12	1.113043	0.312200
WA Application*Fertiliser	6	12	1.607246	0.227810
WA Application*time	3	12	1.889855	0.185143
Fertiliser*time	2	12	0.395652	0.681708
WA Application*Fertiliser*time	6	12	0.302899	0.923569

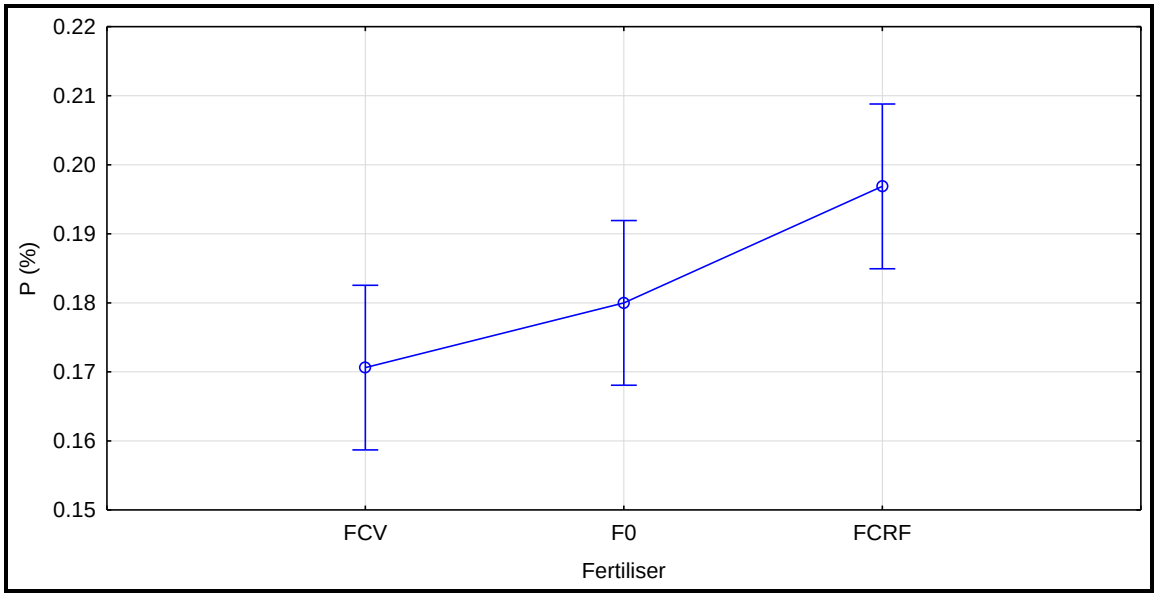

Figure 4.25: Effect of fertiliser variety on foliar P concentration.

K: Statistical significance was found for the effect of time and fertiliser*time interaction, respective *p-values* are presented in Table 4.24. Mean K concentrations were highest at 4 months of age and lowest at 8 months, 1.06 % and 0.97 % respectively. Figure 4.27 graphically illustrates the effect for fertiliser*time interaction on K concentration and significant treatment differences. Mean K concentrations were highest for unfertilised plots at 4 months of age and lowest for F_{CRF} treated plots at 8 months of age, 1.13 % and 0.95 % respectively.

Table 4.24: Foliar K concentration ANOVA results.

Effect	Num. DF	Den. DF	F	P
WA Application	3	12	0.26571	0.848825
Fertiliser	2	12	1.40963	0.281920
time	1	12	17.18149	0.001359
WA Application*Fertiliser	6	12	1.22651	0.358245
WA Application*time	3	12	1.22295	0.343876
Fertiliser*time	2	12	4.75524	0.030143
WA Application*Fertiliser*time	6	12	0.32671	0.910279

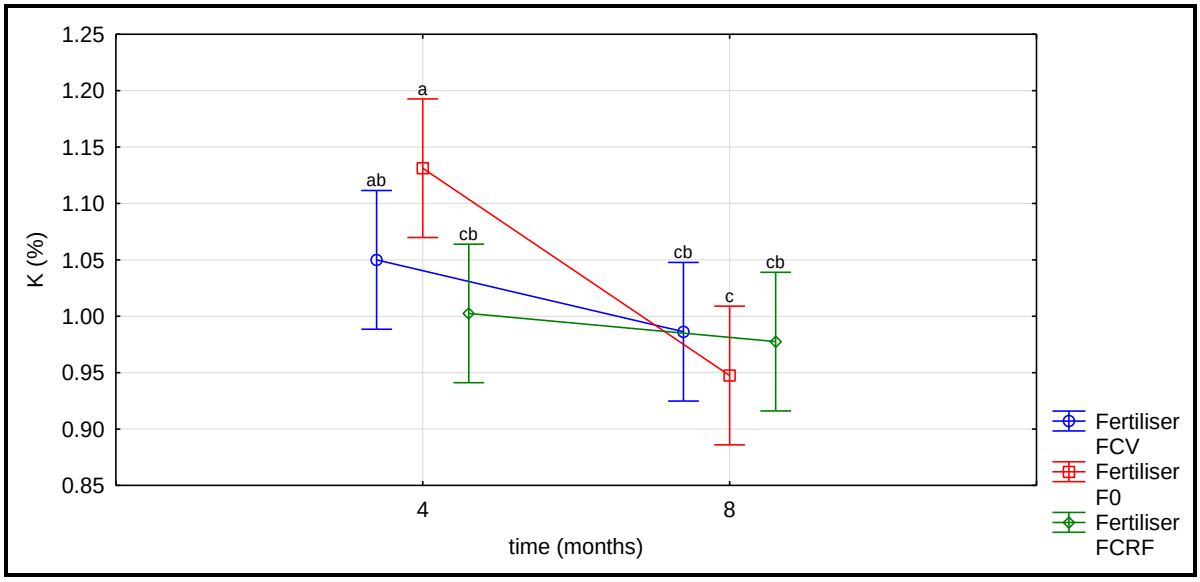

Figure 4.26: Significant treatment differences and effect of fertiliser and time on foliar K concentration.

Ca: Significant treatment differences were found for the effect of fertiliser on foliar Ca concentrations, corresponding *p-values* are presented in Table 4.25. Figure 4.28 graphically illustrates the effect of F_0, F_{CV} and F_{CRF} on Ca concentration and treatment differences. Results showed that the unfertilised plots had the highest mean concentration of 0.77% and F_{CRF} treated plots the lowest concentration of 0.64%. F_{CRF} and F_{CV} treated plots had very similar concentrations; 0.65% for the latter.

Table 4.25: Foliar Ca concentration ANOVA results.

Effect	Num. DF	Den. DF	F	P
WA Application	3	12	0.116369	0.948785
Fertiliser	2	12	7.308357	0.008398
Time	1	12	4.628920	0.052503
WA Application*Fertiliser	6	12	1.122942	0.405167
WA Application*time	3	12	0.955681	0.444916
Fertiliser*time	2	12	0.383286	0.689670
WA Application*Fertiliser*time	6	12	0.373521	0.882091

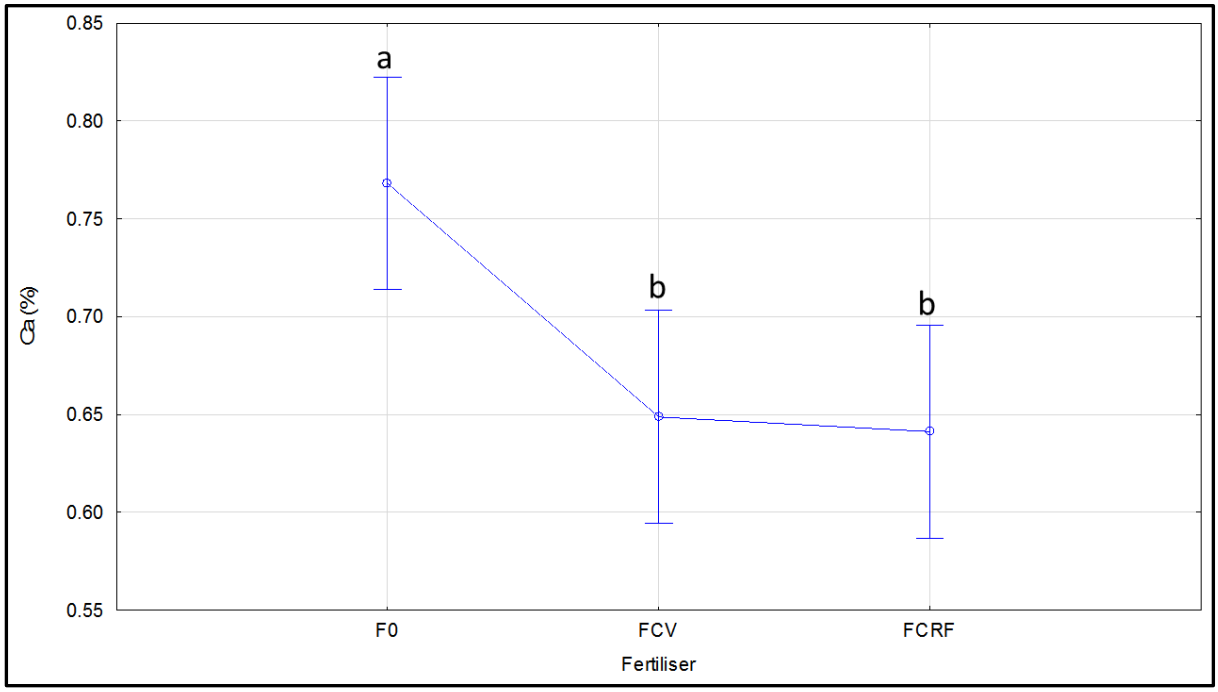

Figure 4.27: Significant treatment differences and the effect of fertiliser on foliar Ca concentration.

Mg: Results indicated significant treatment differences for the effect of fertiliser and fertiliser*time interaction on Mg concentration. The statistical significance and corresponding *p-values* are presented in Table 4.26 and graphically illustrated in Figure 4.29. The highest mean Mg concentration was found for unfertilised plots and the lowest for F_{CV} treated plots, 0.33 % and 0.30 % respectively. Mg concentrations for F_{CV} and F_{CRF} treated plots were very similar, 0.30% for the latter.

Table 4.26: Foliar Mg concentration ANOVA results.

Effect	Num. DF	Den. DF	F	P
WA Application	3	12	1.223129	0.343815
Fertiliser	2	12	8.477551	0.005067
time	1	12	1.800000	0.204550
WA Application*Fertiliser	6	12	0.912925	0.517850
WA Application*time	3	12	0.363265	0.780740
Fertiliser*time	2	12	8.314286	0.005424
WA Application*Fertiliser*time	6	12	1.522449	0.251833

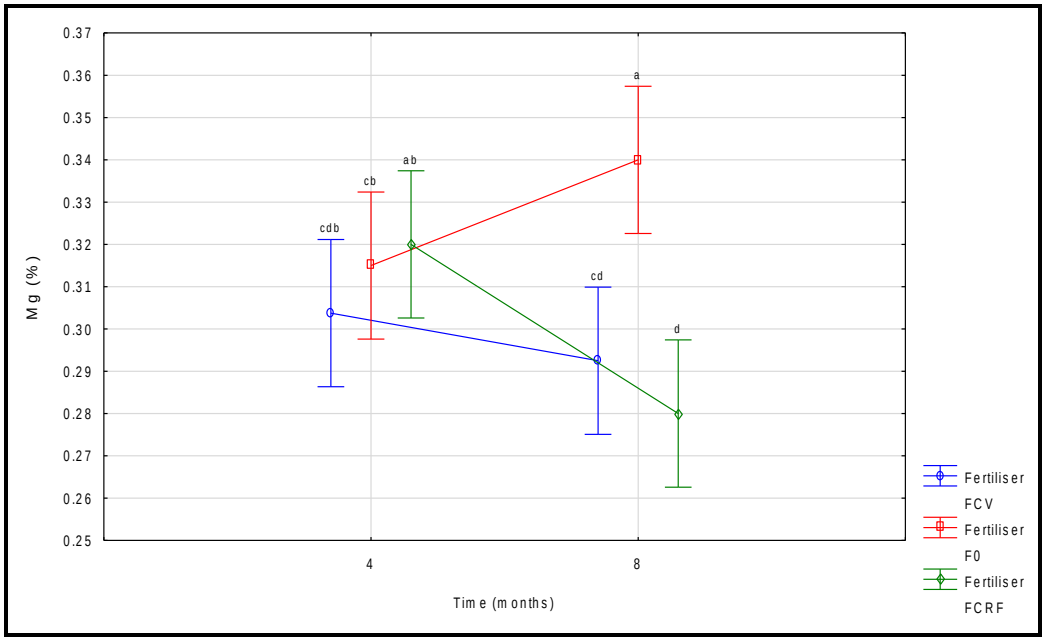

Figure 4.28: Treatment differences and effect of time and fertiliser variety on foliar Mg concentration.

Results showed the highest mean Mg concentration of 0.34% for unfertilised (F_0 treated) plots at 8 months of age. The lowest concentration of 0.28 % was found for plots treated with F_{CRF} at 8 months of age.

Na: Although Na is not an essential element, it can be detrimental to plant growth at high concentrations. Reuter and Robinson (1997) determined acceptable Na concentrations to range between 0.30-0.42 mg/kg for juvenile *Eucalyptus grandis* trees. Results showed significant treatment differences for the effect of fertiliser and time on Na concentrations. Statistical significance for both effects is shown in Table 4.27 and Figure 4.30 graphically illustrates treatment differences and the fixed effect for fertiliser on Na concentration.

Table 4.27: Foliar Na concentration ANOVA results.

Effect	Num. DF	Den. DF	F	P
WA Application	3	12	0.8115	0.511668
Fertiliser	2	12	16.0224	0.000409
Time	1	12	439.2305	0.000000
WA Application*Fertiliser	6	12	0.4459	0.834526
WA Application*time	3	12	0.2727	0.843930
Fertiliser*time	2	12	2.9847	0.088692
WA Application*Fertiliser*time	6	12	1.8580	0.170025

Mean Na concentrations increased significantly from 4 to 8 months of age, 2888.54 mg/kg and 4515.04 mg/kg respectively. The highest mean Na concentration of 4035.13 mg/kg was found for F_0 treated plots and the lowest concentration of 3374 mg/kg for F_{CRF} treatments.

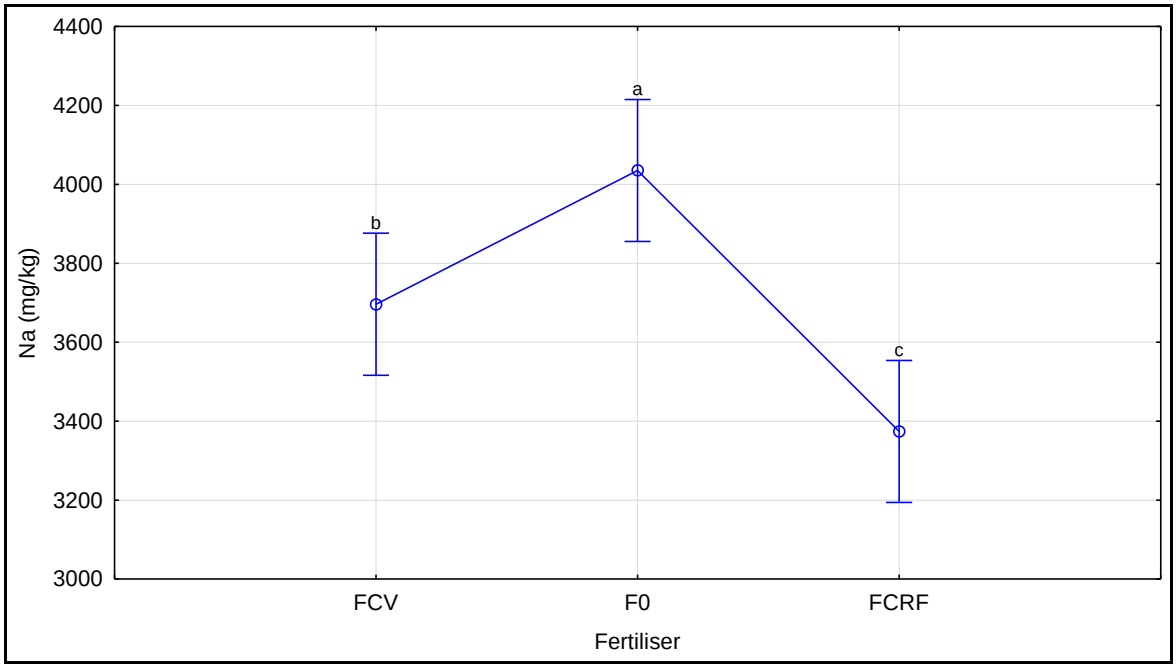

Figure 4.29: Significant treatment differences and the effect of fertiliser on mean foliar Na concentration.

Mn: Results showed statistically significant treatment differences for the effect of time on foliar Mn concentrations. This is presented in Table 4.28. The highest mean Mn concentration was found at 8 months and the lowest at 4 months after trial establishment, 318.96 mg/kg and 249.29 mg/kg respectively.

Table 4.28: Foliar Mn concentration ANOVA results.

Effect	Num. DF	Den. DF	F	P
WA Application	3	12	0.42639	0.737695
Fertiliser	2	12	1.59704	0.242683
time	1	12	73.16750	0.000002
WA Application*Fertiliser	6	12	0.92769	0.509148
WA Application*time	3	12	0.67316	0.584813
Fertiliser*time	2	12	2.22302	0.150911
WA Application*Fertiliser*time	6	12	0.90622	0.521839

Fe: Significant treatment differences were found for the effect of time on foliar Fe concentration. Table 4.29 presents the respective p-value. Results showed the highest mean Fe concentration at 8 months of age and the lowest at 4 months, 60.92 mg/kg and 111.25 mg/kg respectively.

Table 4.29: Foliar Fe concentration ANOVA results.

Effect	Num. DF	Den. DF	F	P
WA Application	3	12	0.2134	0.885167
Fertiliser	2	12	0.5639	0.583353
time	1	12	294.2065	0.000000
WA Application*Fertiliser	6	12	0.9411	0.501364
WA Application*time	3	12	0.4532	0.719796
Fertiliser*time	2	12	0.9655	0.408493
WA Application*Fertiliser*time	6	12	1.1034	0.414632

Cu: Results indicated significant treatment differences for the fixed effect of fertiliser on foliar Cu concentration. This is shown in Table 4.30 with the conforming *p-value* and graphically illustrated in Figure 4.31. The highest mean Cu concentration was found for F_0 treated plots and the lowest for F_{CRF} treatments, 7.56 mg/kg and 5.94 mg/kg respectively.

Table 4.30: Foliar Cu concentration ANOVA results.

Effect	Num. DF	Den. DF	F	P
WA Application	3	12	1.13793	0.373094
Fertiliser	2	12	18.34483	0.000224
time	1	12	1.68966	0.218061
WA Application*Fertiliser	6	12	0.96552	0.487382
WA Application*time	3	12	0.95402	0.445631
Fertiliser*time	2	12	3.86207	0.050711
WA Application*Fertiliser*time	6	12	0.09195	0.995975

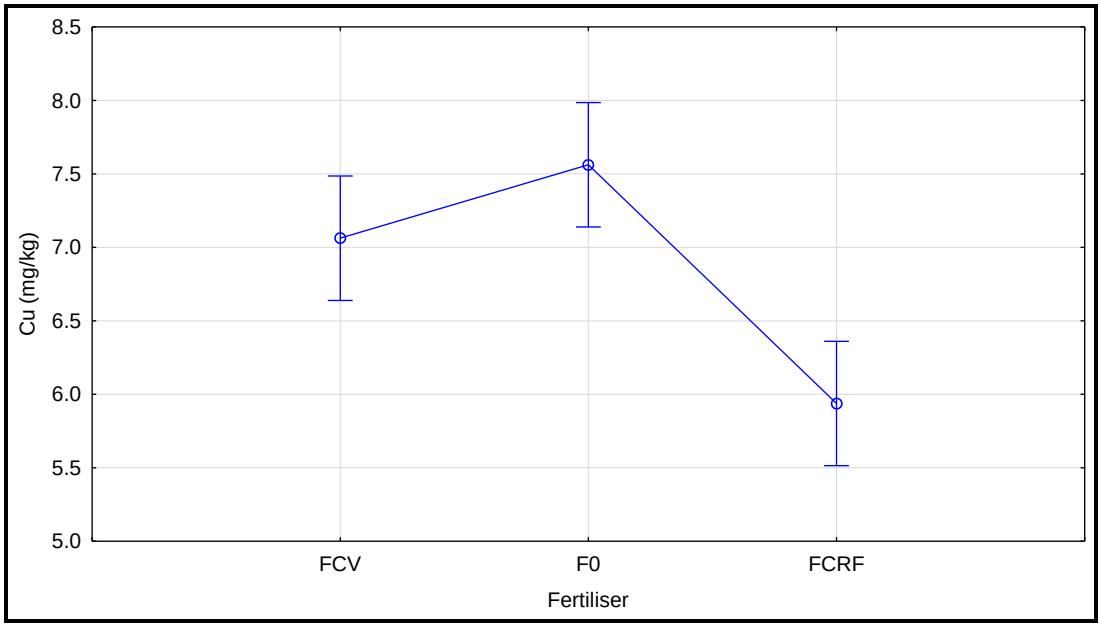

Figure 4.30: Effect of fertiliser on foliar Cu concentration.

Zn: Results showed significant treatment differences for the fixed effects of fertiliser and time on foliar Zn concentrations. Corresponding *p-values* are presented in Table 4.31 and the effect of fertiliser is graphically illustrated in Figure 4.32. Mean Zn concentrations were highest at 8 months of age and less at 4 months, 16.46 mg/kg and 14.33 mg/kg respectively. The highest mean Zn concentration was found for F_0 treated plots and the lowest for F_{CRF} treatments, 15.89 mg/kg and 14.63 mg/kg respectively.

Table 4.31: Foliar Zn concentration ANOVA results.

Effect	Num. DF	Den. DF	F	P
WA Application	3	12	0.56463	0.648645
Fertiliser	2	12	7.12245	0.009137
Time	1	12	53.08163	0.000010
WA Application*Fertiliser	6	12	0.64626	0.692897
WA Application*time	3	12	0.67347	0.584638
Fertiliser*time	2	12	3.00000	0.087791
WA Application*Fertiliser*time	6	12	1.69388	0.205761

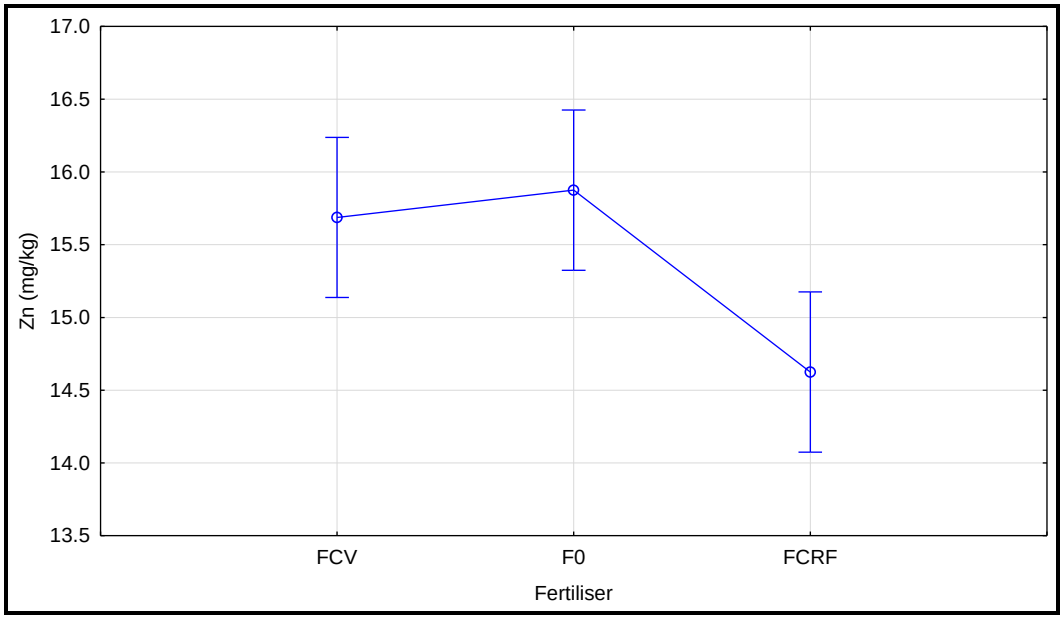

Figure 4.31: Effect of fertiliser variety on foliar Zn concentrations.

B: Results indicated significant treatment differences for the fixed effects of time and fertiliser*time interaction. This is shown in Table 4.32.

Table 4.32: Foliar B concentration ANOVA results.

Effect	Num. DF	Den. DF	F	P
WA Application	3	12	1.2406	0.338127
Fertiliser	2	12	2.7304	0.105364
time	1	12	490.8522	0.000000
WA Application*Fertiliser	6	12	1.5014	0.258184
WA Application*time	3	12	0.0812	0.969011
Fertiliser*time	2	12	5.6870	0.018310
WA Application*Fertiliser*time	6	12	1.4203	0.284339

Mean B concentrations decreased significantly from 4 to 8 months of age; for duration of 4 months the mean value decreased from 38.83 mg/kg to 24.83 mg/kg. The effect of time and fertiliser interaction is graphically illustrated in Figure 4.33. The highest mean B concentration was found for unfertilised plots at 4 months of age and the lowest for unfertilised plots at 8 months of age, 37.88 mg/kg and 22.88 mg/kg respectively.

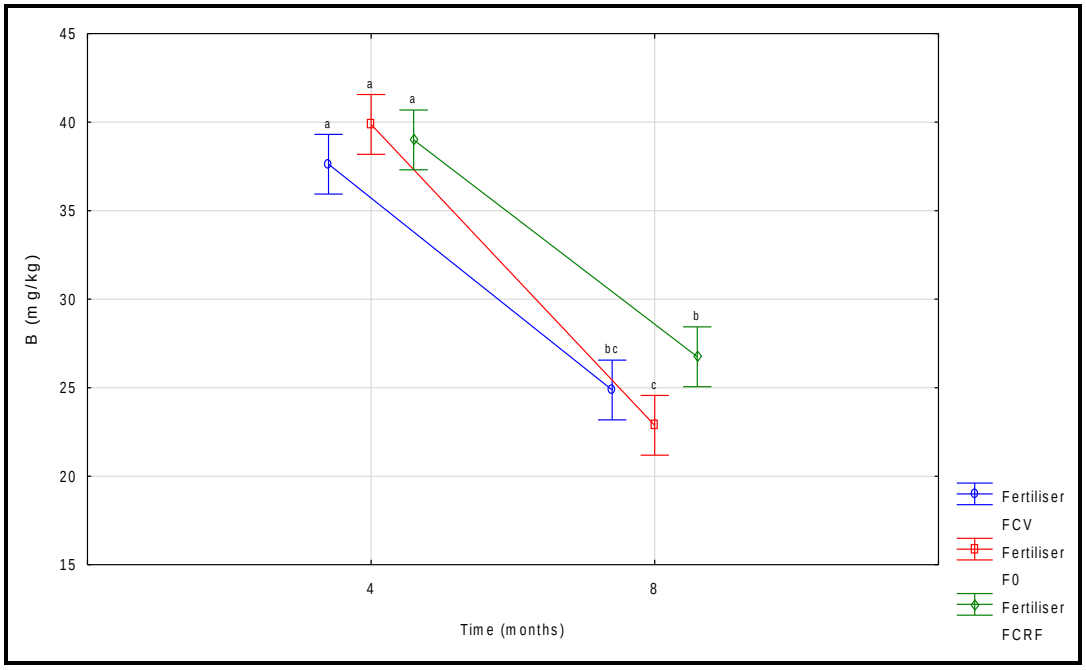

Figure 4.32: Treatment differences and the effect of time and fertiliser variety on foliar B concentration.

4.7.2 Nutrient Ratio

Nutrient concentrations were evaluated according to “optimal values” determined by Linder (1995) using the Nutrient Ratio method. Nutrient status for individual elements was determined by expressing nutrients as a ratio relative to N and comparing the values to the optimal values determined by Linder (1995). Table 4.33 shows foliar nutrients ratios and optimal values at 4 months of age, and shaded blocks indicate elements considered to be sub-optimal. Several and more plots revealed potential nutrient imbalances/deficiencies that might affect growth. Nutrient ratios indicated potential macro-nutrient deficiencies for P and K, due to values falling short of the optimal values 10 and 35. In addition the critical value method revealed sub-optimal nutrient concentrations for P and K. The nutrient ratio method furthermore indicated potential Fe, Cu and Zn imbalances/deficiencies relative to N, values fell short of 0.2, 0.05 and 0.03. The nutrient ratio method identified considerably more plots with nutrient deficiencies relative to the critical values method, and this was attributed to the high N concentrations relative to other macro and micronutrient concentrations.

Table 4.33: Individual nutrients assessed according to the nutrient ratio method and optimal values determined by Linder (1995) at 4 months of age. Shaded blocks indicate elements considered to be imbalanced or sub-optimal. Cells equal to the optimal value, but not shaded, is due to a function of decimal places.

Plot	N	P	K	Ca	Mg	Na	Mn	Fe	Cu	Zn	B	Treatment
4	1.00	5.61	37.54	14.74	10.18	10.67	0.78	0.20	0.02	0.05	0.13	A_0F_0
17	1.00	8.55	52.56	17.95	13.25	12.53	0.91	0.23	0.03	0.07	0.18	A_0F_0
11	1.00	6.62	33.10	14.63	11.15	10.42	1.09	0.22	0.02	0.05	0.14	A_0F_{CRF}
21	1.00	8.06	36.13	13.55	11.29	8.90	0.77	0.21	0.02	0.04	0.14	A_0F_{CRF}
2	1.00	6.00	31.67	14.00	10.00	8.32	0.81	0.22	0.02	0.05	0.12	A_0F_{CV}
15	1.00	5.28	39.62	15.85	10.94	11.53	0.90	0.20	0.03	0.06	0.14	A_0F_{CV}
8	1.00	6.25	40.44	15.44	11.40	11.61	0.79	0.22	0.03	0.06	0.15	A_1F_0
16	1.00	5.53	39.53	16.60	11.86	12.90	0.75	0.22	0.03	0.06	0.14	A_1F_0
10	1.00	6.23	32.46	13.77	11.15	9.70	1.15	0.23	0.02	0.05	0.13	A_1F_{CRF}
24	1.00	6.13	31.94	13.55	9.68	7.90	0.77	0.20	0.02	0.04	0.12	A_1F_{CRF}
7	1.00	5.70	37.64	15.97	10.27	10.05	0.95	0.21	0.02	0.05	0.14	A_1F_{CV}
22	1.00	6.59	43.41	16.28	11.24	11.66	1.07	0.24	0.03	0.06	0.14	A_1F_{CV}
9	1.00	7.63	45.80	16.03	14.12	12.90	1.14	0.24	0.03	0.07	0.17	A_2F_0
19	1.00	6.57	41.24	15.33	11.31	10.73	0.82	0.24	0.03	0.05	0.15	A_2F_0
1	1.00	5.57	28.20	13.77	9.51	7.16	0.76	0.19	0.02	0.04	0.12	A_2F_{CRF}
13	1.00	6.90	34.83	14.48	12.07	9.10	0.86	0.20	0.02	0.04	0.13	A_2F_{CRF}
6	1.00	5.35	35.79	14.05	10.03	9.64	0.82	0.19	0.02	0.05	0.13	A_2F_{CV}
20	1.00	7.38	41.70	15.50	11.44	10.04	0.80	0.22	0.03	0.05	0.14	A_2F_{CV}
3	1.00	6.10	40.34	14.24	10.85	10.85	0.61	0.23	0.03	0.05	0.13	A_3F_0
18	1.00	6.54	43.46	16.15	11.92	11.00	0.84	0.24	0.03	0.06	0.17	A_3F_0
12	1.00	6.01	35.69	14.84	10.25	9.35	1.04	0.19	0.02	0.05	0.13	A_3F_{CRF}
23	1.00	7.80	38.65	14.89	11.35	9.56	0.80	0.22	0.02	0.05	0.14	A_3F_{CRF}
5	1.00	5.73	38.35	15.05	11.11	12.04	1.07	0.25	0.03	0.05	0.13	A_3F_{CV}
14	1.00	6.04	38.49	15.85	13.58	11.57	1.16	0.22	0.03	0.06	0.15	A_3F_{CV}
Opt. Values		10	35	2.5	4	n/a	0.05	0.20	0.03	0.05	0.05	

At 8 months of age, plots were similarly assessed using the “optimal values” determined by Linder (1995). Table 4.34 shows nutrient ratios and optimal values for individual nutrients assessed according to their ratio values using the optimal values.

Table 4.34: Individual nutrients assessed according to the nutrient ratio method and optimal values determined by Linder (1995) at 8 months of age. Shaded blocks indicate elements considered to be deficient or imbalanced. Cells equal to the optimal value, but not shaded, is a function of decimal places.

Plot	N	P	K	Ca	Mg	Na	Mn	Fe	Cu	Zn	B	Treatment
4	1	8.22	43.38	31.05	15.07	22.83	1.51	0.54	0.03	0.07	0.12	A_0F_0
17	1	9.09	44.44	41.92	17.68	25.06	1.49	0.57	0.04	0.09	0.11	A_0F_0
11	1	6.03	27.62	18.41	8.25	12.63	1.05	0.31	0.02	0.04	0.08	A_0F_{CRF}
21	1	6.60	33.66	21.45	8.91	12.62	0.99	0.37	0.02	0.05	0.09	A_0F_{CRF}
2	1	5.56	37.30	27.38	11.51	18.59	1.29	0.45	0.02	0.06	0.11	A_0F_{CV}
15	1	6.61	41.41	29.96	13.66	21.41	1.32	0.48	0.04	0.07	0.10	A_0F_{CV}
8	1	8.19	43.53	29.74	13.36	21.25	1.18	0.44	0.03	0.07	0.09	A_1F_0
16	1	7.80	41.46	61.46	16.59	23.95	1.49	0.74	0.04	0.08	0.11	A_1F_0
10	1	8.19	37.88	24.23	10.92	16.16	1.40	0.38	0.02	0.06	0.09	A_1F_{CRF}
24	1	6.04	31.88	22.82	8.72	12.29	0.97	0.37	0.02	0.05	0.09	A_1F_{CRF}
7	1	7.17	37.54	22.18	9.90	14.83	1.09	0.39	0.03	0.06	0.08	A_1F_{CV}
22	1	6.85	35.62	20.21	9.25	13.53	0.91	0.34	0.02	0.05	0.09	A_1F_{CV}
9	1	8.15	39.48	31.33	13.73	19.81	1.40	0.45	0.03	0.06	0.09	A_2F_0
19	1	7.96	42.92	32.30	14.60	21.09	1.32	0.48	0.03	0.07	0.11	A_2F_0
1	1	5.96	29.47	22.52	9.60	13.30	1.20	0.45	0.02	0.06	0.11	A_2F_{CRF}
13	1	6.64	34.22	22.59	9.30	14.02	0.93	0.33	0.02	0.05	0.09	A_2F_{CRF}
6	1	6.73	35.69	21.89	10.10	15.19	0.95	0.39	0.03	0.06	0.08	A_2F_{CV}
20	1	6.25	35.66	25.37	10.66	16.35	1.10	0.41	0.03	0.06	0.09	A_2F_{CV}
3	1	8.07	41.26	34.53	15.25	21.83	1.23	0.51	0.03	0.08	0.11	A_3F_0
18	1	10.53	51.67	41.15	19.14	27.43	1.93	0.53	0.04	0.09	0.11	A_3F_0
12	1	7.32	36.93	22.30	10.10	15.34	1.22	0.34	0.02	0.06	0.09	A_3F_{CRF}
23	1	6.34	33.21	24.63	10.07	14.25	1.18	0.33	0.03	0.06	0.08	A_3F_{CRF}
5	1	6.12	34.17	24.82	10.79	17.56	1.36	0.44	0.03	0.06	0.09	A_3F_{CV}
14	1	6.25	32.72	28.68	10.66	15.56	1.22	0.40	0.03	0.06	0.09	A_3F_{CV}
Optimal values		10	35	2.5	4	n/a	0.05	0.2	0.05	0.03	0.05	

Results showed nearly all plots were potentially P deficient or at an imbalance apart from plot 18 and could perhaps limit growth; optimal values ranged from 5.56 – 9.09 and were under the optimal value of 10. A number of plots showed potential Cu deficiencies or imbalances, due to optimal values falling short of 0.05. The nutrient ratio method additionally showed potential K deficiencies in several plots and a small risk of Zn deficiencies/imbalances. In addition, the critical value method indicated sub-optimal nutrient concentrations in several plots for K, but excluded the possibility of P and Cu nutrient limitations.

4.7.3 Heavy metals

At 4 and 8 months after trial establishment cadmium, mercury, chromium and lead concentrations were very small for the wood ash application rates of 1200 kg/ha. Table 4.35 shows foliar heavy metal concentrations for selected plots sampled at 4 and 8 months of age. At 4 months of age cadmium was undetectable according to the analysis method described in Section 3.5.5 and mercury, chromium and lead concentrations were in the range of 0.01 – 0.02 mg/kg. At 8 months of age mercury, cadmium, lead and chromium concentrations were similarly very small for the highest wood ash treatments. Cadmium concentrations were once again undetectable according to the test method. Mercury, lead and chromium concentrations ranged between 0.01 - 0.04 mg/kg.

Table 4.35: Foliar heavy metal concentrations for selected plots treated with 1200 kg/ha wood ash at 4 and 8 months of age.

Time	4 months				8 months			
	Cd	Hg	Cr	Pb	Cd	Hg	Cr	Pb
Plot	mg/kg				mg/kg			
3	0	0.01	0.02	0.01	0	0.01	0.04	0
5	0	0.01	0.02	0	0	0	0.04	0.01
12	-	-	-	-	0	0	0.04	0.01
14	0	0.01	0.01	0	0	0.01	0.04	0
18	0	0.02	0.02	0	0	0	0.04	0.01
23	0	0.01	0.02	0	0	0	0.04	0

5. Discussion

5.1 Soil Analysis

5.1.1 Soil pH

In the 8 month monitoring period, wood ash treatments induced a temporary liming effect. Soil analyses at 0, 4 and 8 months showed mean pH values of 5.5, 6.1 and 5.7 and revealed the temporarily liming effect induced by the ash on the whole trial. Although results showed no statistical significance for the effect of individual wood ash application rates on soil pH, there were notable responses. Plots treated with no wood ash (A_0) had a mean pH value of 5.9 at both age intervals and plots treated with the highest ash application rate (A_3) a value of 6.1. Mean soil pH slightly decreased from 5.9 to 5.8 units for 300 and 600 kg/ha application rates.

The large variations in soil pH found at both time intervals were likely a product of previous agricultural lime applications made decades earlier on the site; before the area was converted to commercial forestry. It is unlikely that foresters and commercial forest companies made any lime additions to the soil in recent years, due to liming on coastal sands not being recommended in silviculture. Replications 1 and 2 were located close to the road and the high soil pH variability could possibly have been as a result of agricultural lime additions or the disposal of similar lime materials on the soil several rotations earlier. This likely resulted in greater initial soil pH values, before wood ash treatments were made.

Soil pH increased for approximately 4 months and reverted back to similar values originally found at early trial establishment succeeding the period. Similar results were found by Ohno and Erich (1992a); wood ash incubation studies showed increased soil pH values for the first 25 weeks and a gradual decline as time progressed. A stable pH was achieved at 42 weeks after the experiment was initiated. The increased pH values were expected as wood ash induces a strong alkaline reaction (Demeyer et al., 2001). The decreases at 8 months may well be attributed to the displacement of H^+ and Al^{3+} in the exchangeable sites resultant from Ca, Mg and K additions from the wood ash (Gómez-Rey et al., 2013 and Ring et al., 2006). The soil at the trial site had a low organic carbon content and small cation

exchange capacity, and could possibly have attributed to the leaching of Ca, Mg and K from the soil.

5.1.2 Base cations & CEC

Arenosols characteristically have a low organic matter content and cation exchange capacity relative to other soils (Ngole and Ekosse, 2008). The decreases in total K^+, Mg^{2+} and Na^+ concentrations across all plots after 8 months, illustrated in Figures 4.3, 4.6 and 4.7 in the Results chapter, deviated from most of the wood ash-soil responses described in the literature review. The highest soil base cations concentrations was for Ca^{2+} and may be attributed to the wood ash composition; wood ash contains a significant amount of Ca, as shown in Table 4.1 in Chapter 4. The addition of large quantities divalent Ca^{2+} in the form of wood ash on the soil possibly displaced the monovalent cations Na^+ and K^+, and ions with a smaller mass and ionic radius like Mg^{2+}. Organic carbon greatly affects soil CEC (Rashidi and Seilsepour, 2008; Overstreet, 2014) and can be responsible for up to 80% of the CEC in highly weathered soils (Alleoni, 2008). Alternatively, the significant decrease in organic carbon at 8 months may be attributed to the decrease in CEC observed at the same period. Total units of charge per plot for cationic species was significantly higher at 4 months and lower at 8 months. Untreated wood ash plots had a mean ECEC of 2.79 cmol(+)/kg at 4 months after establishment and 1.84 cmol(+)/kg at 8 months. In addition, the plots treated with the maximum amount of wood ash, 1200 kg/ha, had a greater mean ECEC at 4 months and smaller ECEC at 8 months, 3.19 cmol(+)/kg and 1.96 cmol(+)/kg respectively. The decrease in CEC observed at 8 months was likely as a result of the significant decrease in Mg^{2+} found at 8 months. In addition, the site was burned as a pre-establishment procedure; burning likely temporarily increased the variable charge of the soil and thus the CEC, after 8 months the CEC likely returned back to initial levels before burning was done.

Another contributing factor to the decreased CEC reported at 8 months could be due to obstructed movement of wood ash by the retained slash. Although not likely the case in this project, Gómez-Rey at al. (2012) found that the application of wood ash on vegetation may slow the dissolution of wood ash as a result of decreased soil surface temperatures. The initial application of wood ash on a slash retained site could have delayed the downward leaching of nutrients from the wood ash, and attributed to the decreased concentrations observed in this study.

5.1.3 Organic carbon

Wood ash treatments in combination with an N source can affect soil C and N dynamics during the first months after trial establishment (Gómez-Rey et al., 2013). Soil C decreased from 4 to 8 months. Mean organic carbon content was nearly 50% less at 8 months relative to the prior measurements; this was likely due to the high initial slash load at trial establishment. The effect of wood ash application on organic carbon was insignificant and thus did not provide an accurate indication of whether the results were a direct consequence of the wood ash.

Soil analyses at 4 and 8 months after trial establishment, presented in Appendix 3A and 4A, showed identical soil type classifications and textures. Carbon percentages at both age intervals were however in the range of values obtained in similar sandy soil conditions in KZN, described by Esberg et al. (2010); the authors studied the microbial responses to P additions in six South African soils. Additional factors that could have influenced soil carbon content: The significant decrease could have resulted from the little or no organic carbon input from decomposition of litterfall and harvest residues; the trial remained unplanted for nearly 12 months and was covered in a thick layer of slash in the temporarily unplanted period. In addition, the trial was initially established on a slash retained site and after 8 months slash was still evident and the compartment covered with grassy vegetation after weed control measures were implemented (Figures 4.22 and 4.23). The aerial removal of wood ash shortly after trial establishment could have attributed to the reduced organic carbon values found for this experiment. Saarsalmi et al. (2012) stated that microbial communities are highly responsive to changes in soil pH; the small temporary change in soil pH, described in Section 5.1.1, was less likely to affect soil microbial activity and reduced the rate of decomposition and thus the soil C content.

5.1.4 P & K concentrations

Although there were no statistically significant effects for individual wood ash treatments on soil P anion and K^+ concentrations, the short term nutrient deviations over time were noteworthy. Ash applications on nutrient poor sandy soils can lead to decreased P concentrations (Mandre et al., 2004). P availability can range from 30% to 70% in wood ash (Elliot and Mahmood, 2006) and the large variability in P availability can be attributed to the soil pH buffering capacity (Etiégni et al., 1991a). The trial was established on a coastal arenosol with a low buffering capacity and the

application of loose wood ash increased the soil pH with 0.6 units (maximum value of 6.1) after the first 4 months, this increase was small and led to the reduced availability of P anions. Soil P is fixated by Fe^{2+} and Al^{3+} compounds at pH values smaller than 6 and more favourably by Ca^{2+} and Mg^{2+} at pH values exceeding 7 (Espinoza et al., 2014). The buffering capacity of a soil is central in determining the rise in soil pH from the application of a liming material, and the addition of a liming material in a low buffered soil can reduce P anion availability due to high Ca^{2+} concentrations (Fox et al., 1981). The decreased P concentrations was likely due to the formation of Al^{3+} complexes, as the wood ash contained a significant amount of Al. In addition, P could have leached from the soil given the high leaching ability of a sandy soil. Guerrini et al. (2000) found that the combined application of wood ash and sludge can induce Ca^{2+} : K^{+} soil imbalances and result in acute foliar K deficiencies. It could likely be that the temporary rise in soil pH led to reduced P anion and K^{+} concentrations at 8 months.

The decreased P anion and K^{+} concentrations observed at 8 months could potentially serve as an indication that supplementary P and K sources are needed to offset potential imbalances.

5.2 Survival

Mortality was highest for A_1F_0 treatments at 8 months of age; refer to Table 4.15. A_1F_0 plots were treated with least amount of wood ash and received no supplementary nutrients from fertiliser, and therefore had poor nutrition. The lack of supplementary nutrients from the wood ash and fertiliser explained the poor biomass response found for the treatment, refer to Table 4.19. Poor nutrition led to reduced growth and was unlikely to lead to increased mortality. Contradictory to what was expected, a nearly similar amount of dead seedlings were found for A_0F_{CRF} and A_1F_{CRF} treated plots. These treatments contained the lowest ash rate, the absence of ash and received a large amount of CRF. The experiment tested the application of a substantial quantity (320 g per tree) of polymer coated control released fertiliser containing 80 g N and 20 g P, placed approximately 10cm from the seedling. Although CRF reduces the risk of seedling scorch, the greater mortality in this experiment was possibly a product of the fertilizer being placed too close to the seedling. As fertiliser application on the trial site was operationally implanted, errors

made by the workforce during application could have possibly contributed to the mortality.

5.3 Growth response

Results showed no statistical significance for the main effects of wood ash application rate, fertiliser type and the interaction of both variables on tree growth on a 95 % confidence level. However, the effect of fertiliser type was weakly significant on a 90 % confidence level (p<0.10). The response percentage for individual treatments, presented in Table 4.19 in the Results chapter, ranged between 13 % - 683 % relative to the control. Although the results indicated large growth responses, the responses were weakly significant. This was attributed to the large variation within the trial as a result of mismanaged coppice control. At 4 months of age most seedlings were overshadowed by coppice regrowth and this caused large growth variations within individual plots. For this reason growth data collected at 4 months of age was excluded from the experiment. At 8 months of age, evidence of poor coppice control was still evident as some trees were found growing between multiple stems. The growth response revealed that the application of purely wood ash or fertiliser did not supress growth, bearing in mind the magnitude of the response and weak significance. Moderate wood ash application rates of 0.25 $kg.m^{-2}$ - 0.50 $kg.m^{-2}$ (2. 5 $t.ha^{-1}$ - 5 $t.ha^{-1}$) on nutrient poor sandy soil can increase root biomass and stem growth, but rates exceeding 0.50 $kg.m^{-2}$ (5 t/ha^{-1}) on sandy soils can reduce growth (Mandre et al., 2004).

The application of wood ash and paper sludge combinations on three predominantly sandy soils was found to increase growth in the range of 38 % - 64% relative to chemical fertilisers (Guerrini et al., 2000); experimental conditions given in Section 2.4.3. Eucalypt slash management (burning) is closely representative of the bottom ash used in this trial and previous studies showed that a short term positive growth response could have been expected. Wood ash produced from slash burning can temporarily release large amounts of nutrients and subsequently increase growth (Giardina et al., 2000; du Toit and Dovey, 2005; Gonçalves et al., 2008; du Toit et al., 2008).

5.4 Foliar Analysis

5.4.1 Macronutrients

Foliar nutrient assessment techniques did not indicate severe macronutrient deficiencies across the selected plots at both age intervals. Originally an N limitation was expected, but results showed that the addition of a balancing N source from the CRF and CV fertiliser was able to sustain adequate levels. CRF proved to be superior to CV fertiliser and was able to maintain a higher and slightly more constant N concentration over the experimental period. The critical value method identified 10 plots with sub-optimal P and K concentrations at both age intervals and the nutrient ratio method 61 plots; the nutrient ratio method expressed concentrations as a ratio relative to N and the coastal soils of Zululand are N limited. However, both ratio assessment methods indicated sub-optimal and deficient P and K concentrations and revealed that P and K deficiencies from wood ash applications can be expected. The effects of fertiliser type and time were the only statistically significant effects on P and K concentrations.

The significant decline in mean foliar P concentration between 4 and 8 months was similar to studies done by Ohno and Erich (1994, 1990). Incubation studies by Ohno and Erich (1994) found that wood ash temporarily increases P and K plant availability, but the effects were brief and concentrations declined after the first 25 weeks. In a similar experiment, Ohno and Erich (1990) concluded that the best predictor for P availability from wood ash is based on the level of P in the soil prior to ash application. The decrease in mean K concentrations (refer to Figure 4.27 in Results section) and possible imbalances were similar to the results found by Guerrini et al. (2000). Guerrini et al. (2000) found that the combined application of wood ash and pulp/paper residuals on sandy soils in *Eucalyptus grandis* plantations can induce an acute K deficiency in foliage resultant from Ca^{2+}: K^{+} soil imbalances. Nutrient losses, more specifically P and K in this study, can potentially be attributed to aerial removal of ash during application. The non-significant responses of all macronutrients, excluding N, to the tested wood ash applications rates serves as an indication that up to 1200 kg/ha wood ash can be disposed of on a sandy coastal soil without inducing severe nutrient imbalances. It would appear the application of a supplementary nutrient source (320g CRF, 150g CV fertiliser) or the absence of it

(the unfertilised control) in this experiment, was the main contributing factor for the response observed on all five macronutrients' concentrations in this trial.

5.4.2 Micronutrients and Na

The critical value nutrient assessment method identified a single plot with a sub-optimal Zn concentration at both age intervals and thus did not provide enough evidence to suggest potentially severe micronutrient deficiencies. The nutrient ratio method identified deficient Cu, Zn and Fe levels for 36, 10 and 4 plots respectively, at both age intervals. Due to the large number of plots with potentially deficient Cu and Zn concentrations, identified by the nutrient ratio method, possible causes were investigated.

The foliar nutrient concentration increases observed at 8 months of age for Mn, Zn and B can be explained relative to the temporary liming effect induced by wood ash. Soil pH affects the chemical forms and hence the bioavailability of micronutrients in plants (Dell et al., 2003). Micronutrients are more readily available at slightly acidic soil conditions and less at marginally alkaline conditions, with the exception of Molybdenum (Jensen, 2010). The decreases in soil pH observed several weeks after the wood ash applications most likely increased the bioavailability of micronutrients. In addition to time, the single effect of fertiliser type was statistically significant for foliar Na, Cu and Zn. Concentrations for all three nutrients were significantly higher for unfertilised plots, refer to Figures 4.30, 4.31 and 4.32, and showed that the addition of a supplementary source of nutrition might have affected uptake.

Nutrients taken up by eucalypts often become more diluted as total tree biomass increases over time (Noble, 1991). The decreased macro and micronutrient concentrations observed at 8 months of age can potentially be explained by the translocation of nutrients in the tree with increasing age, and subsequently lead to the dilution of nutrient concentrations. This could explain the significant decreases observed for foliar Cu and Zn at 8 months of age. The biomass growth response for individual treatments, refer to Table 4.19 in the Results chapter, was indicative of possible nutrient dilution. A_2F_0 treated plots had the lowest biomass response of 13% relative to the control and nutrient assessments methods revealed two deficient foliar elements, P and Cu, at 4 and 8 months of age. A_2F_{CRF} treated plots had the greatest growth response of 683% and revealed foliar P, K, Fe, Cu and Zn

deficiencies according to assessment methods. A_1F_{CV} treated plots had an intermediate growth response of 129% and nutrient assessments revealed increased deficiencies for P and Cu at both age intervals relative to the A_2F_0 treatment. Refer to Tables 4.20 and 4.21 for the critical value assessment results and Tables 4.33 and 4.34 for nutrient ratio assessment results. This confirmed the findings of Noble (1991) and showed the likelihood of nutrient dilution with increased biomass accumulation.

5.5 Heavy metals

5.5.1 Soil concentrations

Soil heavy metal concentrations were well below the South African guidelines set by Herselman (2007) as presented in Table 2.5. Chromium was the only element that exceeded the screening values set by the National Environmental Management: Waste Act, 2008 (Act no. 59 of 2008), presented in Table 2.4. All heavy metal concentrations increased significantly from 4 to 8 months after trial establishment. Similar to the foliar analysis; soil cadmium concentrations were nearly undetectable in most plots. This may have been attributed to the solubility of cadmium as a result of the high initial pH values prior to ash application and the temporary increase after. The solubility of heavy metals and trace elements like Cd and Zn increases as soon as pH values decrease below 6.5 and considerably more once pH values decrease to 5.5-6 (Patterson, 2001). The solubility of heavy metals in wood ash is low and as a result uptake by roots is hindered (László et al., 2009).

The bottom ash used in this trial contained a considerable amount of Mn. A review by Pitman (2006) on wood ash use in forestry found that Cd can potentially bond with hydrous Mn and Fe oxides and significantly reduce leaching potential. Given the properties of the sandy soil found at the trial site, this was unlikely the cause. As the trial is located near the industrial town of Richards Bay; a likely explanation for the heavy metal concentrations found in this study could be attributed to the fallout from factories and particles released into the atmosphere and deposited on the soils in the surrounding area.

Heavy metal bioavailability, leaching and toxicity in a soil body are greatly affected by soil pH, the nature of the sorbents, concentration, presence and concentration of organic or inorganic molecules, microbial activity and nutrients (Violante et al., 2010).

The exceptionally low Cd concentrations could be explained by the formation of strong complexes by organic or inorganic molecules with metal ions, due to the organic matter additions from the wood ash and decomposition of the retained slash.

The increases in Hg, Cr and Pb found at 8 months were significant, but their magnitudes were relatively small and likely a result of the decrease in soil pH detected at the same time, shown in Figure 4.10. The decrease in soil pH could have potentially induced a downward movement of heavy metals. Adsorption of Cd in soils is governed by soil pH, organic matter and hydrous oxide content (Pitman, 2006).

5.4.2 Foliar concentrations

Foliar heavy metal concentrations for Cd, Hg, Cr and Pb were less than 1mg/kg at both sampling periods. Metal removal of plants from the soil body is influenced by two main factors. The first is the content of the element in specific above ground plant parts and secondly the total aboveground dry biomass yield (Kacálková et al., 2009). The authors tested Cd, Hg, Cu and Zn uptake generated by an incineration plant for maize and two tree species, willow (*Salix smithiana*) and poplar (*Populus nigra x P. maximowiczii*). Results showed that woody plants had a higher metal uptake relative to maize and the highest Cd concentration of 1.5 - 1.73 mg/kg was found in willow foliage. *Eucalyptus polybractea, Acacia mearnsii, E. cladocalyx* and *Grevillea robusta* are effective heavy metal extraction species due to high biomass accumulation (Mok et al., 2013). The authors tested the heavy metal extraction abilities of several native Australian species from bio solid and soil mixtures using a pot trial after 14 months of growth.

Considering the efficient growth of *Eucalyptus grandis x urophylla* in Zululand conditions and the findings of Mok et al. (2013), the heavy metal concentrations reported by Kacálková et al. (2009) were considerably larger than what was found in this experiment. This showed the heavy metals had a low bioavailability and were possibly affected by the edaphic factors given in Richards Bay.

The analysis of three bottom ash samples representative of the ash used in the trial, shown in Table 4.2 (Chapter 4), showed a maximum Cd concentration of approximately 2 mg/kg. Literature accentuated the serious risk imposed by cadmium, however according to the test method outlined in Section 3.3.3; foliar heavy metal concentrations were nearly undetectable at both time intervals. In addition, the wood

ash analysis found that the highest Pb and Cr concentrations were 2.2 and 11.6 mg/kg. The foliar analyses indicated Pb and Cr concentrations to be smaller than 1mg/kg at both time intervals. High Cd containing wood ash applications on a pine forest soil found no Cd and Pb concentration increases in underlying berry vegetation, concentrations were near normal occurring levels of less than 1mg/kg (Perkiömäki et al., 2003). The small foliar concentrations reported in the experiment were indicative of the diverse bioavailability of specific heavy metals. As, Hg and Pb have a low bioavailability (Laidlaw et al., 2012).

6. Conclusion

The primary objective of this study was to investigate the feasibility of incorporating wood ash, produced from the combustion of biomass, on plantation soils as an alternative disposal method to landfills. In order to achieve this; the soil reaction, nutrient balance and heavy metal content following the application of different levels of ash was closely monitored. The findings of this experiment reported on the soil chemical changes that occurred in the initial months after wood ash applications of 0, 300, 600 and 1200 kg/ha were made. Secondary objectives investigated the effect of the various ash rates on *Eucalyptus grandis x urophylla* growth, with the addition of two supplementary sources of N and P.

The objective of the pilot study was to accurately determine the buffer capacity of the edaphic conditions given by Richards Bay. The buffer capacity was an important variable as it served as an indication of how the soil would react if lime additions were made to it. The buffer capacity was incorporated into a function to determine the lime requirement of the site, refer to Section 3.3.4 in Materials and Methods. A maximum soil reaction depth of 20 cm was incorporated into the lime requirement function, and in effect was crucial in determining the wood ash application rates to be tested in the experiment. The lime requirement was then used to determine the ash requirement, refer to Section 3.3.6 in Material and Methods. As initially expected, the results from the pilot study revealed the site to have a low buffering capacity that is characteristic to sandy soils. The lime buffer capacity was determined as approximately 250 kg/ha and the equivalent ash buffer capacity as 340 kg/ha, using the CCE of the ash. The lowest tested wood ash application rate of 300 kg/ha served as the base ash rate derived from the lime requirement. The base ash requirement

was then doubled and quadrupled in an attempt to push the ability of the soil to accommodate the wood ash.

The findings of this project in cohesion with the pilot study showed that different wood ash application rates in the range of 300-1200 kg/ha, on a poorly buffered sandy soil, can induce a small temporary liming effect. Individual wood ash treatments resulted in smaller pH increases, nonetheless the highest mean soil pH of 6.1 was found for a wood ash application rate of 1200 kg/ha. The effect of time and change in mean soil pH was more central to the obtained results. For an experimental period of 8 months, the tested wood ash application rates increased mean soil pH with 0.6 units after 4 months and returned to similar values reported at 0 and 8 months after trial establishment. The change in soil pH was not as high as initially expected and this is likely a result of the initial uncertainty of the volume of soil that would react with the wood ash. A soil depth of 20 cm was initially selected as the wood ash-soil reaction depth, but the small change in soil pH was an indication that a bigger volume of soil apparently reacted with the wood ash. The findings of this study showed that 1200 kg/ha wood can be safely disposed on a coastal arensol without compromising environmental safety and severely affecting nutrient stability. The highest application rate of 1200 kg/ha was practically a conservative wood ash rate as seen from the findings of this experiment, and most definitely allows for a second wood ash application on the same trial area.

No treatment differences were found for ash additions of 0-1200 kg/ha on heavy metal and nutrient concentrations. However, soil heavy metal concentrations increased significantly over time (from 4 - 8 months), but concentrations were well below toxic levels according to South African and international guidelines. It appeared that the increased concentrations/availability after several months were a product of the temporary shift in soil pH induced by the wood ash. From 4 - 8 months after trial establishment, mean soil Cd concentration increased with 0.01 mg/kg. Mean Hg, Cr and Pb concentrations increased with 0.02, 5.16 and 2.89 mg/kg respectively. Chromium concentrations were considerably higher at 8 months relative to the other elements, but still within the permissible guidelines set by Herselman (2007). Although the concentrations were far below dangerous levels, the results indicated significant signs of heavy metal accumulation in the soil over time. Follow-up wood ash applications are recommended in order to monitor the degree of

soil heavy metal increases relative to the maximum amount of ash that can be applied on sandy soil conditions.

Soil nutrient concentrations were not affected by individual wood ash rates and were a product for the effect of time after the ash applications were made. Soil C, P, K^+, Mg^{2+}, Na^+ and B decreased from 4-8 months, with the exception of Ca^{2+}. The unanticipated mean soil C decrease observed from 4-8 months, 0.39 % - 0.18%, showed that wood ash does not necessarily increase soil organic matter in the topsoil. In an interval of 4 months mean P and K^+ concentrations decreased with 2.58 and 6.46 mg/kg respectively. Mean Mg^{2+} and Na^+ concentrations decreased with 1.67 and 0.46 cmol(+)/kg and Ca^{2+} increased with 1.12 cmol(+)/kg. Although most macronutrient cation concentrations decreased after 8 months, it did not lead to foliar nutrient imbalances or supressed growth. Foliar chemical and visual analyses provided the evidence to show this. Sandy soils characteristically have poor nutrient retention capacity and organic matter content.

The findings of this project showed that foliar heavy metals concentrations, more specifically Cd, Hg, Cr and Pb, from wood ash applications of up to1200 kg/ha does not necessarily become bioavailable and accumulate in high concentrations. At both age intervals foliar Cd was undetectable. Foliar Hg, Cr and Pb concentrations ranged from 0.01 mg/kg - 0.04 mg/kg and were thus substantially lower than 1 mg/kg. Even though the reported foliar heavy metals concentrations were small, the increases were significant and provided some evidence of possible bio-accumulation. Intensive monitoring should be done if a second wood ash application is considered in the near future.

Foliar nutrient analyses and critical value assessment method revealed sub-optimum nutrient concentrations for P, K and Zn at 4 months of age. In addition, several plots indicated sub-optimal K concentrations at 8 months. The foliar analyses of samples collected at both age intervals indicated no severe nutrient deficiencies in any plots and a visual examination of the trial revealed no visual symptoms that suspected nutrient deficiencies. Mean N concentration was the only nutrient significantly affected by time, fertiliser and the interaction of wood ash with both variables. Mean N concentration decreased by 0.17 % from 4-8 months of age. The supplementary 320 g CRF treatment was superior to 150 g CV fertiliser with mean N concentrations

of 2.96 % and 2.74 % respectively. The highest mean N concentration was found for A0FCRF treated plots at 8 months of age and the lowest for plots treated with A0F0 at the same age, 3.09 % and 2.09 % respectively. Mean K concentration decreased from 1.06 % - 0.97 % from 4-8 months of age and mean Zn concentration was highest at 8 months of age and for plots treated with no fertiliser; concentrations were 16.46 and 15.89 mg/kg respectively. Foliar nutrient concentrations were primarily governed by the addition of a supplementary N and P source to the soil.

The biomass index and growth response for treatments (calculated from the BI) showed that the application of wood ash and fertiliser separately or as a combination did not supress *Eucalyptus grandis x urophylla* growth up to 8 months of age relative to the control treatment (A_0F_0). The lowest responses were found for plots treated purely with wood ash: Mean growth responses for A_2F_0, A_1F_0 and A_3F_0 treated plots improved growth with 13 %, 21 % and 48 % respectively, compared to the control. The greatest growth responses were found for plots treated with A_0F_{CRF}, A_0F_{CV} and A_2F_{CRF} which improved by 352 %, 644 % and 683 % relative to the control. The effect for fertiliser type, wood ash and the combination thereof did not dictate the degree of the response. However, the type of fertiliser did significantly affect the height and diameter growth used to calculate the BI. The addition of supplementary N and P sources to the wood ash led to superior growth responses and is recommended if major growth improvements are to be expected from wood ash additions on plantation soils.

7. Limitations and Recommendations

Given the narrow time frame of the project, limitations were unavoidable:

- The wood ash used in the trial was produced from the combustion of untreated *Eucalyptus saligna* waste. The wood ash produced from a pulp and paper mill would likely contain increased amounts of heavy metals and toxic elements. This project tested “clean ash” supposed to wood ash produced by the combustion of treated and mixed materials.
- The experiment was limited to a single soil type and genotype. The reactions of wood ash to different edaphic factors, growing conditions and tree species remain undocumented.
- The soil conditions imposed by the sandy coastal soils in Richards Bay did not allow for greater wood ash application rates for fear of over liming. High initial soil pH values prior to wood ash applications did limit the extent of ash that could have been applied to the trial site.
- The potential downward transmission of heavy metals from wood ash in the soil solution was not included in the scope and remains deduced from the results of previous studies.
- Results were restricted to the first several months after trial establishment and soil chemical changes and growth data was limited to a short time frame.
- The initial wood ash-soil reaction depth was vague and could not be accurately quantified. This placed restrictions on the amount of ash that could be applied to the trial.
- The economic feasibility and cost effectiveness of transporting wood ash from the mill to plantation was not covered in this project. Further research is needed before implementation is made a reality.

It is important a comprehensive chemicals analysis is done on the wood ash intended for soil amelioration, as wood ash applications can potentially damage the environment. Further observation regarding the long term effects of wood ash on soil chemistry and tree growth are recommended. The results obtained from this study reassures the need for future research in the use of wood ash, pulp and paper mill residuals as potential soil ameliorants: The site specific reaction of wood ash on soil chemistry and eucalypt growth seen in this study encourages further research

towards the application of wood ash on a diverse selection of soils and other leading genotypes in the South African forestry industry. The application of wood ash on slightly more acidic soils conditions will allow for a further understanding of the neutralising ability of wood ash.

Future studies should aim to incorporate the monitoring of heavy metals and the likelihood of downward movement into soil water bodies. This will additionally provide an understanding of the degree of downward movement of heavy metals and potential for soil water contamination. The potential for practical implementation of this project is governed by the cost effectiveness of transporting wood ash form the mill to the plantation for disposal. Future research should aim to quantify costs and determine optimum distances that could ensure economic viability.

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Appendix 1A

Foliage nutrient concentrations at 4 months of age assessed according to the critical values method by Dell et al. (1995).

	N	P	K	Ca	Mg	Na	Mn	Fe	Cu	Zn	B
	%					mg/kg					
Plot 1	Adeq.	Adeq.	Sub-opt.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 2	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 3	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 4	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 5	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 6	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 7	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 8	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 9	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 10	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 11	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 12	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 13	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Sub-opt.	Adeq.
Plot 14	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 15	Adeq.	Sub-opt.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 16	Adeq.	Sub-opt.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 17	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 18	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 19	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 20	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 21	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 22	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 23	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 24	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.

*Non-shaded cells = adequate nutrient concentrations; Shaded cells = sub-optimal nutrient concentrations; concentrations are between adequate and deficient levels.

Appendix 2A

Foliage nutrient concentrations at 8 months of age assessed according to the critical values method by Dell et al. (1995).

	N %	P	K	Ca	Mg	Na mg/kg	Mn	Fe	Cu	Zn	B
Plot 1	Adeq.	Adeq.	Sub-opt.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 2	Adeq.	Sub-opt.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 3	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 4	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 5	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 6	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 7	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 8	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 9	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 10	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 11	Adeq.	Adeq.	Sub-opt.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 12	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 13	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 14	Adeq.	Adeq.	Sub-opt.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 15	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 16	Adeq.	Adeq.	Sub-opt.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 17	Adeq.	Adeq.	Sub-opt.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 18	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 19	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 20	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 21	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 22	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 23	Adeq.	Adeq.	Sub-opt.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.
Plot 24	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.	Adeq.

*Non-shaded cells = adequate nutrient concentrations; Shaded cells = sub-optimal nutrient concentrations; concentrations are above deficiency levels, but under adequate nutrient levels.

Appendix 3A

Soil chemical and textural properties at 4 months after trial establishment.

	Soil	Rock	Ph	Resist.	H+	C	P Bray II	K	Ca	Mg	Na	ECEC
Plot nr.		(Vol %)	(KCl)	Ohm	(cmol/kg)	%	mg/kg	cmol(+)/kg				
3	Sand	1	7.7	1880	-	0.57	4	0.11	5.76	0.73	0.07	6.67
4	Sand	1	6.5	3370	-	0.48	9	0.09	1.3	0.4	0.05	1.84
8	Sand	1	7.5	2190	-	0.32	3	0.04	2.39	0.34	0.04	2.81
9	Sand	1	7.3	2490	-	0.44	4	0.06	2.40	0.53	0.05	3.04
16	Sand	1	7.1	2320	-	0.50	4	0.07	3.21	0.54	0.05	3.87
17	Sand	1	7.1	3060	-	0.37	3	0.04	2.30	0.43	0.05	2.82
18	Sand	1	7	2470	-	0.49	4	0.06	2.69	0.46	0.04	3.25
19	Sand	1	6.6	2970	-	0.59	13	0.05	2.46	0.55	0.05	3.11
26	Sand	1	5.7	4940	0.3	0.45	6	0.05	1.00	0.36	0.07	1.48
27	Sand	1	6.3	3710	-	0.38	7	0.07	2.21	0.50	0.05	2.83
28	Sand	1	5.5	5010	0.35	0.17	7	0.08	1.57	0.68	0.08	2.41
29	Sand	1	5.7	5520	0.3	0.28	5	0.05	1.20	0.48	0.05	1.78
43	Sand	1	5.8	2710	0.25	0.32	8	0.06	2.40	0.55	0.06	3.07
44	Sand	1	5.5	3530	0.35	0.20	8	0.09	1.40	0.48	0.05	2.02
45	Sand	1	5.3	4450	0.35	0.25	7	0.05	1.33	0.47	0.05	1.9
47	Sand	1	5.6	5430	0.3	0.30	5	0.05	0.87	0.40	0.06	1.38
49	Sand	1	5.4	3220	0.3	0.39	7	0.07	1.56	0.49	0.06	2.18
50	Sand	1	4.9	3460	0.54	0.70	6	0.07	1.49	0.62	0.06	2.24
56	Sand	1	6.1	2770	-	0.35	8	0.06	2.13	0.56	0.05	2.8
59	Sand	1	5.1	4550	0.4	0.24	5	0.04	0.97	0.39	0.05	1.45
61	Sand	1	5.2	5410	0.3	0.33	7	0.05	1.53	0.44	0.05	2.07
56	Sand	1	6.1	2970	-	0.30	10	0.07	2.11	0.54	0.05	2.77
67	Sand	1	5.5	4610	0.3	0.64	10	0.05	2.44	0.59	0.05	3.13
68	Sand	1	5.9	4400	0.35	0.27	8	0.05	1.07	0.43	0.05	1.6

Appendix 4A

Soil chemical and textural properties at 8 months after trial establishment.

	Soil	Rock	Ph	Resist.	H+	C	P Bray II	K	Ca	Mg	Na	K	ECEC
Plot nr.		(Vol %)	(KCl)	Ohm	(cmol/kg)	%	mg/kg			cmol(+)/kg			
3	Sand	1	6	3970	0.15	0.16	7	0.11	1.61	0.43	0.04	0.05	2.28
4	Sand	1	5.5	5700	0.34	0.12	4	0.09	0.98	0.35	0.04	0.05	1.76
8	Sand	1	5.5	4420	0.34	0.19	3	0.04	1.09	0.26	0.04	0.04	1.77
9	Sand	1	6.2	2730		0.26	4	0.06	1.62	0.28	0.04	0.05	1.99
16	Sand	1	6	4700	0.2	0.23	4	0.07	1.41	0.3	0.04	0.06	2.01
17	Sand	1	5.8	4670	0.25	0.18	5	0.04	0.95	0.3	0.04	0.05	1.59
18	Sand	1	5.7	5150	0.3	0.22	6	0.06	1.14	0.3	0.04	0.05	1.83
19	Sand	1	5.6	4580	0.3	0.17	4	0.05	1.03	0.31	0.04	0.04	1.72
26	Sand	1	5.7	578	0.3	0.16	4	0.05	1.47	0.35	0.04	0.04	2.2
27	Sand	1	5.5	6110	0.3	0.1	3	0.07	0.9	0.32	0.03	0.04	1.59
28	Sand	1	5.5	5820	0.3	0.1	3	0.08	0.8	0.28	0.04	0.04	1.46
29	Sand	1	6	3280	0.2	0.19	3	0.05	1.79	0.32	0.03	0.03	2.37
43	Sand	1	5.9	3840	0.25	0.25	3	0.06	1.74	0.34	0.04	0.04	2.41
44	Sand	1	5.6	5140	0.25	0.15	3	0.09	0.98	0.29	0.03	0.05	1.6
45	Sand	1	5.6	4570	0.34	0.27	4	0.05	1.38	0.38	0.04	0.06	2.2
47	Sand	1	5.5	6800	0.34	0.16	2	0.05	0.82	0.23	0.03	0.04	1.46
49	Sand	1	5.6	5130	0.25	0.17	3	0.07	1.18	0.33	0.03	0.04	1.83
50	Sand	1	5.2	7470	0.3	0.14	3	0.07	0.76	0.27	0.03	0.04	1.4
56	Sand	1	5.6	5990	0.15	0.2	5	0.06	1.28	0.35	0.04	0.05	1.87
59	Sand	1	5.6	6980	0.3	0.17	4	0.04	0.81	0.27	0.03	0.04	1.45
61	Sand	1	5.5	6910	0.3	0.14	5	0.05	0.81	0.27	0.03	0.03	1.44
56	Sand	1	5.9	4280	0.15	0.25	5	0.07	1.45	0.44	0.04	0.05	2.13
67	Sand	1	6	5010	0.2	0.21	5	0.05	1.27	0.38	0.04	0.05	1.94
68	Sand	1	5.9	5910	0.25	0.28	4	0.05	1.1	0.38	0.04	0.05	1.82

Appendix 5A

Soil heavy metal concentrations for selected plots at 4 and 8 months after trial establishment.

	Element (mg/kg)								
	4 months				8 months				Treatment
Plot	Cd	Hg	Cr	Pb	Cd	Hg	Cr	Pb	
4	0.00	0.01	2.04	0.94	0.01	0.05	8.23	4.53	A_0F_0
17	0.00	0.01	1.87	0.89	0.01	0.03	4.92	2.71	A_0F_0
28	0.00	0.00	2.22	0.96	0.01	0.04	7.42	4.11	A_0F_0
43	0.00	0.02	3.28	1.48	0.01	0.01	8.80	4.78	A_0F_0
49	0.00	0.00	3.34	1.38	0.01	0.05	9.32	5.16	A_0F_0
65	0.00	0.01	2.41	0.89	0.01	0.01	8.78	4.4	A_0F_0
8	0.00	0.01	1.62	0.62	0.01	0.04	11.5	5.53	A_1F_0
16	0.00	0.00	1.45	0.73	0.01	0.05	7.8	3.72	A_1F_0
27	0.00	0.00	1.92	1.04	0.01	0.01	4.7	3.00	A_1F_0
47	0.00	0.00	2.26	0.87	0.00	0.01	6.58	3.24	A_1F_0
50	0.00	0.02	2.93	1.47	0.00	0.01	3.70	2.04	A_1F_0
67	0.00	0.00	2.17	0.84	0.01	0.03	10.5	4.95	A_1F_0
9	0.00	0.02	1.73	0.79	0.01	0.03	4.89	2.94	A_2F_0
19	0.00	0.03	2.66	1.72	0.01	0.03	7.54	4.55	A_2F_0
26	0.00	0.02	1.45	0.58	0.01	0.05	8.92	4.59	A_2F_0
45	0.00	0.03	2.41	0.98	0.00	0.03	9.08	4.46	A_2F_0
59	0.00	0.01	2.66	1.09	0.00	0.01	4.64	2.73	A_2F_0
61	0.00	0.00	1.92	0.74	0.01	0.02	7.75	3.62	A_2F_0
3	0.00	0.03	1.16	0.54	0.00	0.01	1.15	0.65	A_2F_0
18	0.00	0.00	2.46	1.11	0.01	0.04	7.52	4.31	A_3F_0
29	0.00	0.00	1.67	0.76	0.00	0.02	4.44	2.46	A_3F_0
44	0.00	0.00	2.36	1.02	0.01	0.04	8.52	4.51	A_3F_0

Element (mg/kg)									
	4 months				8 months				Treatment
56	0.00	0.00	2.55	0.85	0.01	0.05	9.8	4.65	A_3F_0
68	0.00	0.00	2.61	0.87	0.01	0.03	10.5	4.97	A_3F_0
Permissible Levels (mg/kg)	2	0.5	80	56	2	0.5	80	56	

Appendix 6A

Statistical breakdown for height data at 8 months of age.

	Factor level	Factor level	N	Mean	Std. Dev	Std. Err	-95.00%	+95.00%
Units				cm				
Effect								
Total			72	236.01	43.86	5.17	225.71	246.32
WA Application	A_0	18	18	236.87	42.72	10.07	215.62	258.11
WA Application	A_1	18	18	230.00	37.65	8.87	211.28	248.72
WA Application	A_2	18	18	242.49	57.75	13.61	213.77	271.21
WA Application	A_3	18	18	234.69	37.01	8.72	216.28	253.10
Fertiliser	F_{CV}	24	24	239.82	25.75	5.26	228.94	250.69
Fertiliser	F_0	24	24	200.42	36.04	7.36	185.20	215.64
Fertiliser	F_{CRF}	24	24	267.80	39.80	8.12	251.00	284.60
WA Application*Fertiliser	A_0	6	6	244.60	15.45	6.31	228.38	260.82
WA Application*Fertiliser	A_0	6	6	192.47	40.07	16.36	150.42	234.52
WA Application*Fertiliser	A_0	6	6	273.53	17.58	7.18	255.09	291.98
WA Application*Fertiliser	A_1	6	6	246.00	14.47	5.91	230.82	261.18
WA Application*Fertiliser	A_1	6	6	197.33	44.52	18.18	150.61	244.06
WA Application*Fertiliser	A_1	6	6	246.67	26.58	10.85	218.77	274.56
WA Application*Fertiliser	A_2	6	6	242.40	46.18	18.85	193.94	290.86
WA Application*Fertiliser	A_2	6	6	198.93	35.67	14.56	161.50	236.37
WA Application*Fertiliser	A_2	6	6	286.13	58.07	23.71	225.19	347.08
WA Application*Fertiliser	A_3	6	6	226.27	12.93	5.28	212.70	239.83
WA Application*Fertiliser	A_3	6	6	212.93	28.93	11.81	182.57	243.30
WA Application*Fertiliser	A_3	6	6	264.87	43.68	17.83	219.03	310.70

Appendix 7A

Statistical breakdown of DBH data at 8 months of age.

	Factor level	Factor level	N	Mean	Std. Dev	Std. Err	-95.00%	+95.00%
Units				cm				
Effect								
Total			72	1.72	0.57	0.07	1.58	1.85
WA Application	A_0		18	1.85	0.74	0.17	1.49	2.22
WA Application	A_1		18	1.64	0.41	0.10	1.44	1.84
WA Application	A_2		18	1.74	0.65	0.15	1.42	2.06
WA Application	A_3		18	1.64	0.42	0.10	1.43	1.85
Fertiliser	F_{CV}		24	1.74	0.49	0.10	1.54	1.95
Fertiliser	F_0		24	1.28	0.34	0.07	1.14	1.43
Fertiliser	F_{CRF}		24	2.12	0.52	0.11	1.90	2.34
WA Application*Fertiliser	A_0	F_0	6	2.06	0.81	0.33	1.21	2.92
WA Application*Fertiliser	A_0	F_{CRF}	6	1.18	0.45	0.18	0.71	1.65
WA Application*Fertiliser	A_0	F_{CV}	6	2.31	0.38	0.15	1.92	2.71
WA Application*Fertiliser	A_1	F_0	6	1.79	0.20	0.08	1.58	2.01
WA Application*Fertiliser	A_1	F_{CRF}	6	1.28	0.40	0.16	0.86	1.70
WA Application*Fertiliser	A_1	F_{CV}	6	1.84	0.37	0.15	1.45	2.22
WA Application*Fertiliser	A_2	F_0	6	1.64	0.35	0.14	1.28	2.00
WA Application*Fertiliser	A_2	F_{CRF}	6	1.26	0.28	0.11	0.97	1.55
WA Application*Fertiliser	A_2	F_{CV}	6	2.31	0.75	0.31	1.52	3.09
WA Application*Fertiliser	A_3	F_{CV}	6	1.48	0.20	0.08	1.27	1.69
WA Application*Fertiliser	A_3	F_0	6	1.41	0.24	0.10	1.15	1.66
WA Application*Fertiliser	A_3	F_{CRF}	6	2.04	0.47	0.19	1.54	2.54